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COMMERCIAL PRODUCTION OF BRANDIES

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COMMERCIAL PRODUCTION OF BRANDIES¹

M. A. JOSLYN² AND M. A. AMERINE³

INTRODUCTION

THE COMMERCIAL PRODUCTION of table and dessert wines in California has been discussed in Bulletins 639 and 651.⁴ In the present publication the production of brandies from wines by distillation is considered. Beverage brandies are grape spirits sold at a proof of 100° or below for use as a beverage. Fortifying brandies are grape spirits of 160° to

TABLE 1
ALCOHOLIC STRENGTHS BY VARIOUS SYSTEMS

| United States | Great Britain | France and Belgium at 15°C | Italy and Russia at 15.6°C | Germany | Cartier system |
|---------------------|---------------------|----------------------------|----------------------------|------------------------|----------------|
| <i>proof spirit</i> | <i>proof spirit</i> | <i>volume per cent</i> | <i>volume per cent</i> | <i>weight per cent</i> | |
| 5.7 | 5* | 2.8 | 2.9 | 2.3 | — |
| 11.4 | 10 | 5.6 | 5.7 | 4.6 | — |
| 22.8 | 20 | 11.3 | 11.4 | 9.2 | — |
| 34.4 | 30 | 17.1 | 17.2 | 13.9 | — |
| 57.3 | 50 | 28.4 | 28.6 | 23.5 | — |
| 85.8 | 75 | 42.7 | 42.9 | 35.9 | — |
| 100.0 | 87.5 | 49.8 | 50.0 | 42.5 | — |
| 114.2 | 100 | 56.9 | 57.1 | 49.3 | 10.1? |
| 142.8 | 125† | 71.3 | 71.4 | 63.9 | 26.7 |
| 171.3 | 150 | 85.5 | 85.6 | 80.1 | 33.9 |
| 194.3 | 170 | 97.0 | 97.2 | 95.3 | 41.5 |
| 200.0 | 175.08 | 100.0 | 100.0 | 100.0 | 44.0 |

* Or 95 under proof.

† Or 25 over proof.

Source of data:

Simmonds, C. Alcohol. 574 p. (See p. 275.) Macmillan and Co., Ltd., London, England, 1919.

189° proof intended for use in the fortification of wines or for sale for other legal purposes. Spirits distilled at 190° proof or over are designated as "spirits-fruit" or "fruit spirits"; they may not legally be called "brandy."

"Proof" is an old English term for the strength or quality of spirits. In England 100 per cent proof spirit contains 57.1 per cent by volume of alcohol. In the United States, proof spirit, or 100° proof spirit, con-

¹ Received for publication May 27, 1941.

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⁴ Amerine, M. A., and M. A. Joslyn. Commercial production of table wines. California Agr. Exp. Sta. Bul. 639:1-143. 1940.

Joslyn, M. A., and M. A. Amerine. Commercial production of dessert wines. California Agr. Exp. Sta. Bul. 651:1-186. 1941.

tains 50 per cent by volume of alcohol, and 200° proof spirit is absolute alcohol. The legal United States definition is as follows:

Proof spirit is held and taken to be that alcoholic liquor which contains one half its volume of alcohol of a specific gravity of 0.7939 at 60° F referred to water at 60° F as unity. Proof spirit has at 60° F a specific gravity of 0.93426 (*in vacuo*), 100 parts by volume of the same consisting of 50 parts of absolute alcohol and 53.73 parts of water. The difference of the sum of the parts of alcohol and water and the resulting 100 parts of proof spirit is due to the contraction which takes place when alcohol and water combine.⁵

A comparison of the alcoholic strengths as given by the various systems in use in different countries is shown in table 1. The problems of the California beverage-brandy industry are many. The quality of the wines used for distillation could be improved; pomace wines or wines of low quality make the production of the best-quality potable brandy difficult. The rational operation of the stills under existing government regulations is difficult; since these regulations, which have been adopted to insure protection of the revenue, greatly restrict the distiller's freedom of operation. There is need for wider knowledge of the factors influencing efficient still operation. Finally, the present economic status of the industry and the competition of the product with other distilled spirits are complicating factors.

Although the production of wines for distillation purposes is briefly considered here, a more complete discussion of the methods of making table wines is found in Bulletin 639. The use of fortifying brandy for fortification purposes is treated in Bulletin 651. The present bulletin is based on investigations of the divisions of Fruit Products and Viticulture, as well as on observations of the best current distillery practices.

⁵ United States Bureau of Internal Revenue. Gauging manual embracing instructions and tables for determining the quantity of distilled spirits by proof and weight. 579 p. (Quotation from p. 41.) United States Government Printing Office, Washington, D.C. 1939.

ECONOMIC STATUS OF THE CALIFORNIA BRANDY INDUSTRY^a

Official data on brandy production and consumption in the United States, shown in tables 2 and 3, do not segregate grape brandy but cover all fruits. In California practically all brandy (99 per cent) is grape brandy, whereas in other states brandy is made largely from other fruits.

As shown in table 2, the consumption of fruit brandy as a beverage

TABLE 2

UNITED STATES PER-CAPITA CONSUMPTION OF ALCOHOLIC BEVERAGES, YEARS
BEGINNING JULY 1, AVERAGES 1909-1913 AND 1935-1939

| Period average | Wine | | | Distilled spirits | | | Malt liquors |
|-------------------|----------------|----------------|----------------|--------------------------|--------------------------|--------------------------|-----------------------------|
| | Total | Dessert | Table | Total | Brandy* | Other | |
| | <i>gallons</i> | <i>gallons</i> | <i>gallons</i> | <i>proof gallons</i> | <i>proof gallons</i> | <i>proof gallons</i> | <i>gallons</i> [†] |
| 1935-1939..... | 0.77‡ | 0.34 | 0.43‡ | 0.79 | 0.02 | 0.77 | 12.65 |
| 1909-1913..... | 0.52 | 0.20 | 0.32 | 1.48 | 0.03 | 1.45 | 20.77 |

* Includes all fruit brandy used for purposes other than fortification of wine.

† Converted from barrels at 31 gallons per barrel.

‡ Includes homemade table wine in 1935-1939 of 0.25 gallons; commercial table wine amounts to 0.18 gallons and total commercial wine 0.52 gallons.

Sources of data:

Compiled by S. W. Shear, Giannini Foundation of Agricultural Economics, College of Agriculture University of California.

1909-1913 from: Shear, S. W., and G. G. Pearce. Supply and price trends in the California wine-grape industry, Part 2. Univ. California Giannini Foundation Mimeo. Rept. 34. tables 3 and 7. June, 1934.

1935-1939:

Wine data from: Shear, S. W. Deciduous fruit statistics as of January, 1941. Univ. California Giannini Foundation Mimeo. Rept. 76: 72. 1941.

Other data are United States imports for consumption plus tax-paid withdrawals from bonded warehouses; malt liquors also include tax-free withdrawals consumed on premises. Import data from releases of United States Bureau of Foreign and Domestic Commerce. Withdrawals from releases of Bureau of Internal Revenue, Alcohol Tax Unit.

has been less since Repeal than before Prohibition. It averaged 2,414,000 gallons⁷ during 1935-1939 as compared with 3,195,000 gallons during 1909-1913, or 0.02 gallon per capita as compared with 0.03. The consumption of beverage fruit brandy produced in the United States averaged only 0.02 gallon per capita, which is small in comparison with that of other distilled spirits (0.77 gallon). Consumption and production of

^a Prepared in collaboration with S. W. Shear, Associate Agricultural Economist in the Experiment Station and Associate Agricultural Economist on the Giannini Foundation, who supplied the statistical data used. General references on this subject will be found on p. 73.

⁷ A wine gallon in the United States contains 231 cubic inches. A wine gallon of 100° proof spirits is known as a proof gallon. A wine gallon of 180° proof spirits is therefore said to be 1.8 proof gallons. For a discussion of the proof gallon and its use in taxation, see Henderson. (Henderson, Y. The proof gallon: a federal tax strait jacket. Quarterly Journal of Studies on Alcohol 2:46-56. 1941.)

California brandy, in general, paralleled one another until the peak production in 1938. The increased production in that year, was due largely to the brandy made for the Growers Grape Products Association from the grape surplus.

Fortifying brandy, made almost exclusively from grapes in Califor-

TABLE 3
CALIFORNIA PRODUCTION OF FRUIT BEVERAGE AND OF FORTIFYING BRANDY AND
UNITED STATES CONSUMPTION OF FRUIT BEVERAGE BRANDY,
YEARS BEGINNING JULY 1, 1933-1940

| Year beginning July 1 | California production of brandy* | | United States consumption of beverage brandy | | |
|-----------------------|----------------------------------|-------------------------------|--|-------------------------------|-------------------|
| | Fortifying | Beverage | Total | California brandy†† | |
| | | | | Quantity | Per cent of total |
| | 1 | 2 | 3 | 4 | 5 |
| | <i>thousand proof gallons</i> | <i>thousand proof gallons</i> | <i>thousand proof gallons</i> | <i>thousand proof gallons</i> | <i>per cent</i> |
| 1933..... | 4,711 | 2,324 | 1,477§ | 1,180 | 79.9 |
| 1934..... | 8,034 | 1,258 | 1,893 | 1,457 | 77.0 |
| 1935..... | 17,606 | 1,627 | 2,151 | 1,526 | 70.9 |
| 1936..... | 10,543 | 1,402 | 2,650 | 1,713 | 64.6 |
| 1937..... | 18,920 | 2,314 | 2,407 | 1,270 | 52.8 |
| 1938..... | 17,055¶ | 9,812¶ | 2,390 | 1,236 | 51.7 |
| 1939..... | 16,303¶ | 1,380¶ | 2,460 | 1,164 | 47.3 |
| 1940..... | 21,570 | 2,500 | 2,657 | 1,359 | 51.1 |

* An average of about 99 per cent of California fruit brandy is grape brandy.

† Includes annual average exports of 1933-1939 of less than 500 proof gallons.

‡ Some California brandy moves in and out of bonded warehouses in other states, so that disappearance from California plants as given may not all be consumed in the years indicated.

§ Involves estimates.

¶ Includes in 1938, 8,887,000 proof gallons of beverage brandy and 4,610,000 proof gallons of fortifying brandy, and in 1939, 150,000 proof gallons of beverage brandy and 84,000 proof gallons of fortifying brandy, all produced under the 1938 grape-prorate program.

|| Preliminary estimates.

Sources of data:

Compiled by S. W. Shear, Giannini Foundation of Agricultural Economics, College of Agriculture, University of California.

Cols. 1, 2, and 4 based on: Shear, S. W. Deciduous fruit statistics as of January, 1941. Univ. California Giannini Foundation Mimeo. Rept. 76. Grape table 16, cols. 2, 6, and 9, and comparable data for 1940; except that for col. 9 in 1938-1940, revised beverage brandy production data as reported by Wine Institute, March 19, 1941, were substituted.

Col. 3: United States tax-paid withdrawals from bonded warehouses as reported by the United States Bureau of Internal Revenue plus United States brandy imports from the United States Bureau of Foreign and Domestic Commerce.

nia, is ten times greater in amount than beverage brandy. It is used almost entirely in the production of dessert wines.

The diversion of surplus raisin and table grapes to the wine industry and the economic consequences of this diversion are discussed in Bulletin 651.

THE COMPOSITION OF BRANDIES AND THE FACTORS INFLUENCING IT⁸

ALCOHOLS⁹

The alcohols are the most important volatile substances present in the wine which are carried over into the brandy. They are important not only because of their influence on the taste, but also because they constitute the largest fraction of the product and have important physiological effects.

*Ethyl.*¹⁰—Ethyl alcohol, or ethanol ($\text{CH}_3\text{CH}_2\text{OH}$), is a colorless liquid. It has a slight odor and a burning taste. Pure ethyl alcohol burns with a pale-blue, practically nonluminous flame. Its boiling point has been variously reported, but 172.9° F (78.3° C), at 760-mm pressure, is usually taken as the best figure. It solidifies at about -178.6° F (-117° C). It is lighter than water, having a specific gravity of 0.79384 at 60° F referred to water at 60° F as unity, when measured *in vacuo* (0.79359 in air). The thermal coefficient of expansion is large, about three times that of water.

Ethyl alcohol is slightly hygroscopic and is miscible with water in all proportions. There is an increase in total volume and an increase in temperature when ethyl alcohol and water are mixed. But when the mixture is returned to the original temperature, there is a contraction in total volume. Both the heat evolved on mixing and the final decrease in volume indicate that a hydrate is formed.

The contraction varies with the relative amounts of alcohol and water: calculated as a percentage of the sum of the initial volumes, the contraction is at a maximum of 3.64 per cent when the mixture contains three molecules of water to one of alcohol. This occurs when 53.94 volumes of alcohol are mixed with 49.84 volumes of water; the 103.78 volumes of water and alcohol become reduced (after the mixture is cooled to the original temperature) to 100 volumes. Expressed in another way, the maximum contraction is obtained by mixing 52 volumes of alcohol with 48 volumes of water, the volume of the resulting mixture being 96.3 at 20° C instead of 100. When increasing amounts of water are added to a definite volume of alcohol and the actual contraction in volume is determined, the position of the maximum contraction is different, a maximum being reached when there are eight molecules of water to one of alcohol.

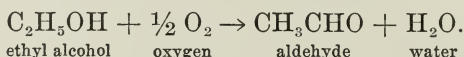
⁸ General references on this subject in addition to those mentioned in specific footnotes in the section will be found on p. 73.

⁹ "Alcohol" is derived from the Arabic *al-koh'l*. It was originally applied to antimony sulfide, later to fine powders, and finally to spirits.

¹⁰ Where the word "alcohol" is used alone in this bulletin, it refers to ethyl alcohol.

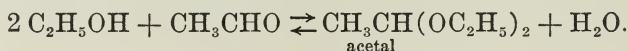
Ethyl alcohol forms a constant-boiling-point mixture (azeotropic mixture) with water at about 97.4 per cent alcohol (see p. 33). Similar mixtures are formed with benzene and other organic compounds. Ethyl alcohol is not only completely miscible with water, but it also is a good solvent for many substances: esters, essential oils, fatty acids, and others. Water-alcohol mixtures are therefore very satisfactory solvents.

Ethyl alcohol is oxidized to aldehydes and acetic acid. Although this ordinarily takes place very slowly under distillery aging conditions, the running of air into the still will increase the aldehyde content:

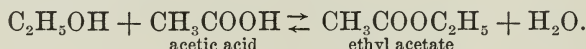


Simmonds¹¹ reports that charcoal, especially in the presence of ferrous salts, in alcohol markedly increases the aldehyde content. Ferrous salts also act as catalysts for oxidation of alcohol by strong oxidizing agents.

Alcohol slowly reacts with aldehydes to produce acetals:



It also reacts with acids to yield esters:



The physiological effects of ethyl alcohol have been studied by numerous workers. It has narcotic as well as stimulating properties. In general, its effect is on the nervous system, although Sollmann¹² recognizes that in favorable doses it increases the flow of gastric juice, saliva, and bile.¹³ Various studies¹⁴ have shown that it has a definite food value, that it definitely is burned, and that the energy is used for bodily functions.

¹¹ Simmonds, C. Alcohol. 574 p. (See especially p. 161-62.) Macmillan and Co., Ltd., London, England. 1919.

¹² Sollmann, T. A manual of pharmacology. 5th ed. 1,190 p. W. B. Saunders Co., Philadelphia, Pa. 1936.

¹³ For a more recent discussion of the influence of ethyl alcohol on the digestive tract see Beazell and Ivy. (Beazell, J. M., and A. C. Ivy. The influence of alcohol on the digestive tract. Quarterly Journal of Studies on Alcohol 1(1):45-73. 1940.) These authors consider that the effect of alcohol on the formation of bile has been inadequately studied. They agree, however, that in optimum doses ethyl alcohol stimulates the production of saliva, gastric juice (particularly the acid), and pancreatic juice and that it inhibits hunger contractions. The increase in appetite generally ascribed to alcoholic beverages they attribute to the stimulation of the end organs of taste and general sensibility, or to a central effect, such as a promotion of a feeling of well-being. Direct experimental evidence on this latter point is difficult to obtain. (Tennent, D. M. Factors influencing the effects of alcohol on blood sugar and liver glycogen. Quarterly Journal of Studies on Alcohol 2(2):263-70. 1941.)

¹⁴ Richter, C. P. Alcohol as a food. Quarterly Journal of Studies on Alcohol 1(4):650-62. 1941.

Mitchell, H. H., and E. G. Curzon. The food value of ethyl alcohol. Quarterly Journal of Studies on Alcohol 1(2):227-45. 1940.

More recently the relations of chronic alcoholism to what Jolliffe¹⁵ calls "alcoholic polyneuropathy" and to vitamin B₁ deficiency have been extensively studied. This also has led to the suggestion of the possible fortification of potable spirits with vitamins. The physiological and medicolegal aspects of this problem are too numerous for discussion here.

As far as the pharmacological use of the various potable spirits is concerned, ethyl alcohol is the active agent. Haggard, Greenberg, and Cohen¹⁶ have shown, however, that the amount of alcohol in the blood depends on the type of alcoholic beverage consumed and whether before or after a meal: after a meal larger quantities of all alcoholic beverages can be consumed before the concentration of alcohol in the blood is increased to a given level.

The production of ethyl alcohol in fermentation has already been considered in Bulletin 639. The amount of alcohol present in the finished brandy will depend on the method of distillation, on the extent of reduction in proof by the addition of water, and on the type and length of the aging process. Usually it varies in beverage brandies from 40 to 55 per cent—80° to 110° proof—though bottled brandies rarely contain more than 50 per cent.

Methyl.—Methyl alcohol (wood alcohol, methanol, CH₃OH) is a colorless liquid. It burns with a pale-blue flame and has a slight spirituous odor and a burning taste. It boils at 150.8°F (66° C). Its specific gravity is 0.79647 at 59° F (15° C).

The physiological properties of methyl alcohol have been the subject of much study. It is no more toxic than ethyl alcohol to isolated tissues, and the general acute symptoms of excessive use of it resemble those of ordinary alcoholic intoxication. The chief difference is that it has a stronger local irritation than ethyl alcohol and the after-effects are more protracted and serious—incurable blindness from retrobulbar neuritis and retinitis being a frequent feature of methyl alcohol intoxication, according to Sollman (cited in footnote 12, p. 8).¹⁷ The sources and amounts present in potable spirits are therefore of some interest.

Grapes of high pectin content are more likely to yield methyl alcohol

¹⁵ Jolliffe, N. Vitamin deficiencies and liver cirrhosis in alcoholism. *Quarterly Journal of Studies in Alcohol* 1(3):517–57. 1940.

¹⁶ Haggard, H. W., L. A. Greenberg, and L. H. Cohen. Quantitative differences in the effects of alcoholic beverages. *New England Journal of Medicine* 219:466–70. 1938.

See also: Haggard, H. W., L. A. Greenberg, and G. Lolli. The absorption of alcohol with special reference to its influence on the concentration of alcohol appearing in the blood. *Quarterly Journal of Studies on Alcohol* 1:684–726. 1941.

¹⁷ See also: Haggard, H. W., and L. A. Greenberg. Studies in the absorption, distribution and elimination of alcohol. IV. The elimination of methyl alcohol. *Journal of Pharmacology and Experimental Therapeutics* 66:479–96. 1939.

on fermentation, especially if they are moldy and high in tartaric acid. Methyl alcohol is commonly believed to arise from the hydrolysis of pectin. Espinosa¹⁸ suggests that high temperature of the still leads to hydrolysis of the pectin and release of methyl alcohol. Wines made by a fermentation on the skins would therefore be expected to have a greater methyl alcohol content. Hartmann,¹⁹ however, reports that some California white wines do not show the expected low methyl alcohol content as compared with red wines, possibly owing to the fact that, in this state,

TABLE 4
ORGANOLEPTIC AND CERTAIN PHYSICAL AND PHYSIOLOGICAL
CHARACTERISTICS OF THE HIGHER ALCOHOLS

| Alcohol | Formula | Source | Odor and taste | Relative toxicity* |
|-----------------------|--|--------------|-----------------------------------|--------------------|
| <i>n</i> -propyl..... | C ₂ H ₅ CH ₂ OH | Fermentation | Spirituos, slight burning | 2.5 |
| Isopropyl.... | CH ₃ CHOHCH ₃ | Fermentation | Spirituos, slight burning | — |
| <i>n</i> -butyl..... | C ₂ H ₅ CH ₂ CH ₂ OH | Fermentation | Not agreeable, slight burning | 3.8 |
| Isobutyl..... | (CH ₃) ₂ CHCH ₂ OH | Fermentation | Strong, irritating, burning | — |
| <i>n</i> -amyl..... | CH ₃ (CH ₂) ₃ CH ₂ OH | Isoleucine | Disagreeable, fusel, very burning | — |
| Isoamyl..... | (CH ₃) ₂ CHCH ₂ CH ₂ OH | Leucine | Disagreeable, fusel, very burning | 5.0 |
| <i>n</i> -hexyl..... | CH ₃ (CH ₂) ₄ CH ₂ OH | —† | Not disagreeable | — |
| <i>n</i> -heptyl..... | CH ₃ (CH ₂) ₅ CH ₂ OH | —† | Characteristic§ | — |
| <i>n</i> -octyl..... | CH ₃ (CH ₂) ₆ CH ₂ OH | —† | Pleasant, fruity§ | — |
| <i>n</i> -nonyl..... | CH ₃ (CH ₂) ₇ CH ₂ OH | —† | Pleasant, fruity§ | — |
| <i>n</i> -decyl..... | CH ₃ (CH ₂) ₈ CH ₂ OH | —† | Pleasant, fruity§ | — |

* Compared to methyl alcohol as 1.0.

† Reported as a product of fermentation in the acetone-butyl alcohol fermentation. See: Marvel, S. C., and A. E. Broderick. The composition of yellow oil obtained in the manufacture of *n*-butyl alcohol by fermentation. American Chemical Society Journal 47:3045-51. 1925.

‡ Said to come from fermentation but may also be derived from other sources—destructive distillation of lees, for example, or possibly from the reduction of the corresponding acids produced from the fats.

§ Each has a characteristic pungent smell in pure 100 per cent solution. In dilute solutions their odor is more agreeable.

white wines are frequently made by a partial fermentation on the skins, particularly white wines destined for distillation.

Valaer²⁰ reports from a trace to a small percentage of methyl alcohol in seventy-two samples of California brandy taken from government-supervised warehouses. He also suggests that brandies made with neutral spirits as a base are not likely to show methyl alcohol, while nongrape brandies may be detected by their higher methanol content. The average content in his grape brandies was less than 0.05 per cent.

Wines obtained from grapes fermented on the skins and pomace dis-

¹⁸ Espinosa, N. A. Methanol in alcoholic beverages. III. Origin of methanol. Asociación Química Argentina Anales 19:39-70. 1931. Abstracted in: Chemical Abstracts 26:798. 1932.

¹⁹ Hartmann, B. G. Report on wines. Association of Official Agricultural Chemists Journal 20:149-51. 1937.

²⁰ Valaer, P. Brandy. Industrial and Engineering Chemistry, industrial edition 31:339-53. 1939.

tillates should show an increased methyl alcohol content. Pirani,²¹ for example, found over ten times as much methyl alcohol in distillates obtained from grape pomace as in those obtained directly from wine. In one grape-pomace brandy he found 4.0 per cent. Careful operation of stills in removing the "heads" should reduce this difference (see p. 49).

TABLE 5
PERCENTAGE COMPOSITION OF FUSEL OIL FROM VARIOUS SOURCES

| Source | <i>n</i> -Propyl <i>per cent</i> | <i>n</i> -Butyl <i>per cent</i> | Isobutyl <i>per cent</i> | <i>n</i> -Amyl* <i>per cent</i> | Isoamyl* <i>per cent</i> | Residue <i>per cent</i> |
|---|-------------------------------------|------------------------------------|-----------------------------|------------------------------------|-----------------------------|----------------------------|
| Brandy: | | | | | | |
| Sample 1 ^a | 11.7 | 63.8 | 0.0 | 24.5 | — | 0.0 |
| Sample 2 ^b | 11.9 | 49.3 | 4.5 | 34.3 | — | 0.0 |
| Sample 3 ^c | 12.1 | 0.0 | 2.9 | 85.0 | — | —† |
| Fermented sugar-yeast extract ^d | 3.7 | 0.0 | 2.7 | 93.6 | — | 0.0 |
| Whiskey: | | | | | | |
| Sample 1 ^e | 20.4 | — | 23.9 | 14.6 | 36.3 | 4.8 |
| Sample 2 ^e | 1.7 | — | 12.2 | 23.4 | 59.7 | 3.0 |
| Potato spirit: | | | | | | |
| Sample 1 ^f | 6.9 | — | 24.4 | 68.8 | — | 0.04 |
| Sample 2 ^f | 3.9 | — | 16.5 | 79.5 | — | 0.14‡ |
| Fermented molasses ^g | 13.0 | — | 42.0 | 36.0 | — | 9.0 § |

* *n*-amyl and isoamyl alcohols not separated except for whiskey.

† Hexyl and heptyl alcohols demonstrated.

‡ Hexyl alcohol.

§ Ethyl alcohol.

Sources of data:

^a Ordonneau, Ch. Sur la composition des eaux-de-vie de vin. [Paris] Académie des Sciences Comptes Rendus 102:217-19. 1886.

^b Claudon, E., and E.-Ch. Morin. Sur la présence de l'alcool butylique normal dans une eaux-de-vie de Cognac; comparaison des alcools supérieurs de cette eau-de-vie avec produits dans la fermentation du sucre par la levure elliptique [Paris] Académie des Sciences Comptes Rendus 104: 1187-89. 1887.

^c Morin, E.-Ch. Sur la composition chimique d'une eau-de-vie de vin de la Charente-Inférieure. [Paris] Académie des Sciences Comptes Rendus 105:1019-22. 1887.

^d Claudon, E., and E.-Ch. Morin. Produits de fermentation du sucre par la levure elliptique. [Paris] Académie des Sciences Comptes Rendus 104:1109-11. 1887.

^e Schicktan, S. T., A. D. Etienne, and W. I. Steele. Analysis of fusel oil by azeotropic distillation. Industrial and Engineering Chemistry, analytical edition 11:420-22. 1939.

^f Windisch, K. Ueber de Zusammensetzung der Brantweine. Arbeiten aus dem Kaiserlichen Gesundheitsamte 8:140-228. 1892.

^g Bell, J. Report of the Select Committee on Spirits. House of Commons, Great Britain. p. 93. 1891

But the prevalent use of grape pomace for preparing distillery material in California is a complicating factor.

Higher Alcohols.—Numerous homologues of methyl and ethyl alcohol, commercially known collectively as "fusel oil," are present in brandy. The more important of these are listed in tables 4 and 12.

The proportions of the various higher alcohols present in brandy and

²¹ Pirani, F. Analisi de acquaviti di vino e di vinaccia di spiriti grezzi e rettificati. Annali Chimica Applicata 17:176-87. 1927.

other spirits are not at all constant (table 5). This variation is probably due to the differences in the composition of the raw material, to the use of yeasts of various types and degrees of purity, to the extremely variable fermentation conditions, to the method of distillation, and to various other factors. In addition, the analytical methods used by some of the earlier workers for determining the various higher alcohols is open to question. The total amount of the higher alcohols in brandy seldom exceeds 0.3 per cent, so that they constitute less than 1 per cent of the total alcohol content. In the early investigations, *n*-butyl alcohol was found to be the predominant higher alcohol in brandy. Valaer, however, found that amyl alcohol is present in greater amounts. He did, however, find a larger percentage of propyl and butyl alcohol in French brandy than in California brandy. Trost²² has likewise found that amyl alcohol predominates. Valaer was unable to find heptyl or hexyl alcohols in the "heads" and "tails" from a California distillery—in the quantity, 4 liters, taken. Trost, using a spectrographic method, found heptyl alcohol and smaller amounts of isobutyl and isohexyl alcohols. As table 5 indicates, amyl alcohol is the predominant higher alcohol in spirits produced from corn, potatoes, and rye. An unexpectedly high isobutyl alcohol content is reported in a rum from molasses. As far as brandy is concerned, comparative analysis of spirits produced from raw materials of known composition, with a uniform method of fermentation and distillation, would do much to elucidate the problem of the relative proportions of the higher alcohols present, and the factors which influence their formation.

It is now well known that some of the higher alcohols in fermented beverages are produced from the amino acids. Leucine, for example, yields inactive amyl alcohol and isoleucine yields active amyl (*d*-amyl) alcohol. If sufficient ammonium salts are present, the production of higher alcohols is reduced because the yeast assimilates ammonium salts preferentially.

Although the toxicity of the higher alcohols is somewhat greater than that of ethyl alcohol, experiments to show that the toxicity of certain potable spirits is due to their higher-alcohol content have usually failed.²³ The nauseating odor of fusel oils may be a factor in their undesirability, but the actual amounts present are so small that reported toxicity from this source must usually be discredited.²⁴ Ethyl alcohol is the most im-

²² Trost, F. *Sugli alcooli superiori dei cognacs*. *Annali di Chimica Applicata* 25:660-68. 1935. *Abstracted in*: *Chemical Abstracts* 30:6503. 1936.

²³ Allen, for example, took whiskey containing 2 per cent added higher alcohols every evening for a period of two months without headaches or other ill effects. (Allen, A. H. *The chemistry of whiskey and allied products*. Society of Chemical Industry Journal 10:305-14. 1891.)

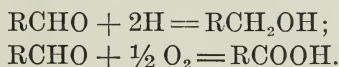
²⁴ See p. 679 in Sollmann (cited in footnote 12, p. 8). This author suggests that the sinister reputation of spirits containing a larger percentage of higher alcohols

portant substance present in spirits from the physiological point of view, chiefly because there is so much of it compared to all of the other constituents.

ALDEHYDES

Acetaldehyde.—Acetaldehyde is the most important aldehyde present. The presence of as much as 0.1 per cent acetaldehyde in raw whiskeys has been reported,²⁵ but the amount present in normal brandies is appreciably less. Acetaldehyde is a colorless, inflammable, and very light liquid (specific gravity 0.780 at 68° F), boiling at 69.8° F (21° C); hence during distillation it is one of the first constituents to distill. Propionaldehyde and iso- and *n*-butyraldehyde likewise boil at temperatures lower than alcohol. Acetaldehyde is miscible in all proportions with water and alcohol.

On reduction, aldehydes yield alcohols, and on oxidation, the corresponding acids:



On exposure to air, acetaldehyde slowly takes up oxygen and forms acetic acid. The production of acetals by the reaction of an alcohol and an aldehyde has already been mentioned.

The physiological importance of aldehydes in brandies is small, since they are present in such small quantities. Even the 0.1 per cent reported above would probably produce only a local irritation, according to Sollmann.

Acetaldehyde is produced in normal alcoholic fermentation. Greater quantities would be expected in fermentations where sulfur dioxide is present to fix the aldehyde as it is formed.

Furfural.—Furfural is a colorless liquid having a density greater than water, 1.1598 at 68° F relative to water at 39.2° F as unity. It boils at 243.1° F (161.7° C). It is only fairly soluble in water but is miscible with alcohol in all proportions.

The amounts found are small (traces to 2.0 mg per liter), except in spirits produced from grape pomace, in fire-heated pot stills, or in spirits stored in charred barrels, where larger amounts may be found, up to 7.0 mg per liter. (See p. 19 and 22.)

The physiological importance of amounts of furfural of the order of those present in spirits is slight; large doses, however, are convulsive.²⁶

is due to their generally higher percentage in the cheaper spirits, which are more likely to be consumed in larger quantities and in debauches. The question of possible after effects of the higher alcohols on the human system should also be considered.

²⁵ Doran, J. M., and G. F. Beyer. Character of moonshine liquor. American Journal of Public Health 13:831-34. 1923.

²⁶ McGuigan, H. Action of furfural. Journal of Pharmacology and Experimental Therapeutics 21:65-75. 1923.

ESTERS

Esters are substances formed by the nonionic reaction of alcohols and acids. They are hydrolyzed by boiling with dilute acids or alkalis or by prolonged heating with steam. The rate of esterification is increased by raising the temperature. According to the mass-action law, the percentage conversion of the acid or alcohol will depend on the relative molal concentrations of each. These factors are apparently of some importance in still operation when high-acid wines are distilled, though conclusive experimental evidence of the practical importance of this is lacking. Some esters are always found in wines, and ethyl acetate, at least, may be formed by direct bacterial action. This is the chief ester found in brandy. Not only are the other esters of the common acids and alcohols in brandy present in small quantities, but their contribution to the aroma is limited. More complex esters, however, may be important. Esters of propyl and butyl alcohols have been identified. In the fractionation of French and California brandies, Valaer found amyl acetate in the latter but not in the former. This ester he believes may be produced by the multiple refluxing of the acetic acid in the presence of amyl alcohol in continuous stills. Its bananalike aroma is not considered desirable in brandies. Methyl anthranilate is found in wines made from certain varieties of *Vitis labrusca* and similar American grapes, but these are seldom, if ever, used for distillation, and this ester would probably be destroyed by distillation.

The nature and the origin of oenanthic ether (ester) reported by various European workers is somewhat obscure.²⁷ Ordonneau²⁸ considered this impure fraction to be mainly responsible for the character of cognac, although he thought it to be a terpene.

Valaer isolated an oil from cognac which he considers to have the character of cognac. He was also able to isolate this from dry wine lees, and he believes the principal difference between California and French brandy to be that the latter is distilled in fire-heated pot stills with the lees, whereas California brandies are distilled in continuous stills from low-acid wines. Pomace wash, rather than wine, is used for distillation in California, but usually for fortifying brandy.

The minimum amount of esters which should be present in genuine brandy has been generally set at 40 mg per liter of 100°-proof spirits.

²⁷ Commercial "oenanthic" ether is very impure ethyl *n*-heptylate (ethyl enanthate). It has a very pronounced aroma and has been used in making imitation brandies. Ethyl caproate, caprylate, pelargonate, and caprate are also used for this purpose as well as amyl caproate and isoamyl butyrate and mixtures of these and other esters. The commercial oil of cognac is apparently produced from lees and contains a large percentage of these esters in addition to other substances.

²⁸ Ordonneau, Ch. Sur la composition des eaux-de-vie. [Paris] Académie des Sciences Comptes Rendus 102:217-19. 1886.

Amounts less than this are occasionally reported but most undiluted samples conform to this standard. No particular harmful physiological reactions of the esters have been reported in the concentrations found in brandies. Amyl acetate, when inhaled in large quantities, has some toxic properties, however.

ACIDS

Acetic and lactic acids are produced in normal yeast fermentations in minimum amounts and by bacterial action in larger amounts. Acetic acid may also result from the oxidation of acetaldehyde. Acetic and lactic acids are weak acids and contribute little to the character of a brandy unless present in large amounts, over 100 mg per liter of 100°-proof spirits.

No important direct organoleptic or physiological properties can be ascribed to these acids, though they have an indirect influence in reactions with alcohols, as has been previously mentioned, particularly acetic acid. The presence of inorganic acids in brandy will be discussed later. During aging some acid material is derived from the wood.

OTHER CONSTITUENTS

The nature and properties of the other substances found in wines have been discussed in Bulletins 639 and 651.

The presence of ammonia and various organic bases should be noted. These are partially, at least, due to the destruction of yeast cells during distillation. Some of these contribute to the disagreeable odor of newly distilled brandies.

SIGNIFICANCE OF ANALYTICAL TESTS

After the period when phylloxera attacked the vineyards of France, the price of brandies advanced, and there was a great temptation for falsification of neutral spirits to give them the character of grape brandy. This was particularly true in Germany but was also prevalent in France until the grape acreage and brandy stocks were restored about the beginning of the present century.

The detection of falsified brandies by organoleptic examination was one means of combatting this situation. (See p. 63.) Later, chemical analysis was tried as a means of determining the authenticity of spirits. The most important test devised for this purpose was to determine the esters, acids, aldehydes, furfural, and higher alcohols. The summation of these constituents must, according to the French law, exceed 280 mg per liter of 100 per cent alcohol.²⁹ This sum is called the "nonalcohol coefficient." The purpose of this law is apparently to prevent highly rectified spirits, particularly nonvinous spirits, from being flavored and

²⁹ Flanzy, M., and M. Lamazon-Betbeder. Sur la composition des eaux-de-vie de la région languedocienne. *Annales de Technologie Agricole* 1:106-19, 1938.

then diluted and sold as beverage brandy. Flanzky and Lamazon-Betbeder criticize the minimum-limit law as penalizing vacuum-distilled and other highly rectified but authentic grape brandies. It does, however, give a measure of the important constituents of the spirit and prevents many frauds. Genuine California beverage brandies easily exceed this minimum. In Belgium a maximum limit has also been fixed in order to prevent poorly distilled brandies from reaching the market.

Lusson³⁰ divides the nonethyl alcoholic constituents into two groups: (1) oxidation products, acids, and aldehydes; (2) esters and higher alcohols. The concentration of the constituents in the first group varies with age and is assumed to be of importance in evaluating the age of brandies.

FACTORS INFLUENCING THE COMPOSITION OF BRANDIES

Substances from the Grapes.—Both desirable and undesirable constituents are derived from the grapes. Furthermore, the composition of the grape will have both direct and indirect effects on the composition of the wine and hence on the spirits. Grapes grown in calcareous soils are considered to yield wines of enhanced aroma. The prices paid for wines for distillation in the Cognac district of France are found to vary according to the composition of the soil on which the grapes used for the wine were grown.³¹ Those grown on the most calcareous soils (Grande Champagne district) receive an advanced price as compared to those produced in the slightly calcareous soils at the edges of the Cognac district (270 francs per hectoliter as against 200 in 1893), while grapes grown in certain soils in the south of France are said to yield wines which result in "earthy"-tasting spirits.

Moldy or mildewed flavors may result from poor grapes and will be transmitted to the spirits, sometimes even when special precautions are taken in distilling the wine. Wines of high acidity are said to form more esters during distillation, as previously mentioned.

Red wines are not so desirable for brandy production as white wines. The reasons for this are not clear. Either the longer fermentation on the skins extracts more undesirable substances, or red grapes naturally contain larger amounts of substances undesirable from the standpoint of brandy production. Possibly hydrolysis products of the pigments, tannins, or constituents found in the bloom may prove inimical to the quality of brandy. Markley, Sando, and Hendricks,³² for example, found

³⁰ Lusson, F. Composition de eaux-de-vie. *Moniteur Scientifique* 10:785-88. 1896.

³¹ Baudoin, A. Les eaux-de-vie et la fabrication du Cognac. 278 p. Librairie J.-B. Baillière et Fils, Paris, France. 1893.

Delamain, R. Histoire du Cognac. 141 p. Librairie Stock, Paris, France.

³² Markley, K. S., C. B. Sando, and S. B. Hendricks. Petroleum ether-soluble and ether-soluble constituents of grape pomace. *Journal of Biological Chemistry* 123(3): 641-54. 1938.

the bloom to yield on hydrolysis, palmitic, stearic, oleic, linoleic, and oleanolic acids as well as other acids, hydrocarbons, and unidentified resinous substances. Fermentation on the skins for a long period of time may be expected to extract more of the bloom. The distillation of pomace, in either pot or continuous stills, may increase this extraction. Grinding of the pomace before distillation will also release the oils of the seeds. Theron,³³ in writing on the methods for improving South African beverage brandies, strongly recommends the use of only white grapes, immediate pressing after crushing, distillation of the lees with the wine in pot stills, and no sulfur dioxide nor musts of high sugar concentration.

Among the most important substances in the brandies which are derived from grapes are the varietal aromas, particularly the muscat aroma. The nature of these substances is unknown, but their influence on the character of certain California brandies is very marked.

Substances Obtained in Fermentation.—Sulfur dioxide added to musts during fermentation may persist in the wines and will then be distilled. During aging it will be oxidized to sulfuric acid, an objectionable constituent of fine beverage brandies. The presence of sulfur dioxide or sulfuric acid in fortifying brandy is less serious, since it will be largely diluted out in fortification. But fortifying brandy stored or aged in iron tanks may become excessively contaminated with metal, and the wine fortified with it may show an abnormally high metallic content. Fortifying brandy of high sulfur dioxide content should not be stored; for if it is, it usually has to be redistilled before use because of the excessive pickup of metals from metal pipes or containers.

Free sulfur from the dusts applied to grapes during the growing season may be reduced to hydrogen sulfide, an evil-smelling, volatile substance, if the lees are left in contact with the wine too long or under too warm conditions after fermentation. This compound is objectionable not only because of its own odor but also because organic sulfur compounds, known as mercaptans, may be formed. These compounds have a nauseating odor if allowed to distill over. A part of the disagreeable odor of newly distilled brandy is due to these substances. If present in only small amounts, they are dissipated during aging.

Wines fermented at high temperatures show reduced production of desirable volatile fermentation products (or less retention of them), and, in addition, may yield an undesirable "heated" taste to the brandy.

The original sugar content will also influence the quality of the brandy. Of two wines with the same percentage composition of volatile constituents, the wine of lower alcohol content will yield a brandy having

³³ Theron, D. de Wet. South African brandy. *Wine and Spirit* 11(97):4399, 4401. 1939.

a greater percentage of volatile substances, *if* both are distilled at the same proof.

Wines made from diluted pomace are very low in total acid and high in pH. Such wines, if of the same alcohol content and if distilled at the same proof, will show less aroma and have a less desirable aroma than those made from undiluted musts.

So far as the nitrogen content of the must influences the type of fermentation and the production of higher alcohols, it is a factor in the composition of the brandy. Kayser and Demolon,³⁴ however, consider that the volatile materials found in the grape, as well as those formed during the aging of the wine, are much less important to the quality of the brandy than those formed during fermentation. The use of different cultures (either pure or mixed) is therefore a possible method of changing the quality of the brandy. Certain organisms other than wine yeasts are very active in forming higher alcohols.

Of the constituents of the wine found in brandy, the most important ones derived from fermentation are: ethyl alcohol; higher alcohols such as propyl, butyl (normal and iso), amyl, hexyl, heptyl, and octyl; acetaldehyde; acetic and other acids; and ethyl acetate and other esters. Bacterial action in the wine before distillation may markedly increase the concentration of acids and ethyl acetate.

The percentage of alcohol by volume in the new brandy, as distilled, ranges, as we have mentioned, from 70 to about 90. This is, of course, soon reduced by dilution with distilled water to a constant figure of about 51 per cent, in the case of California beverage brandies. In new beverage brandy, the higher alcohols vary from 100 to 500 mg or higher per liter of 100 per cent alcohol, according to the higher-alcohol content of the original wine and the method of operation of the still. The aldehydes range from 10 to 500 mg per liter; the acids (as acetic) from 10 to 200; and the esters from 40 to 300. (Some furfural also is formed during distillation.)³⁵

Substances Produced during the Distillation.—The question of whether the lees from the fermentation should be added to the wine before distillation is important. Ordonneau³⁶ many years ago found in the lees a substance boiling at 173°–175° C, which was one of the principal constituents of the bouquet. Valaer has also produced odorous substances by distilling the lees. (See p. 14.) The current practice with cognac is to add the lees of the *same* fermentation tank to the wine prior to dis-

³⁴ Kayser, E., and A. Demolon. Contribution à l'étude des eaux-de-vie de vin. *Revue de Viticulture* 28:119–22. 1907.

³⁵ Graff, G. Weinbrand, seine Verfälschungen und deren Nachweis. *Zeitschrift für Untersuchungen der Lebensmittel* 54:102–37. 1927.

³⁶ Ordonneau, Ch. *Reported by*: Baudoin, p. 53. (Cited in footnote 31, p. 16.)

tillation. The distillation of 100 per cent lees, however, is a different matter. The high concentration of solid matter, or, if diluted, the low concentration of alcohol relative to the volume of the lees, are new variables which influence the composition of the product. In general, material containing more than 20 per cent lees is considered to yield spirits differing from wine brandies in pot-still distillations. The method of operation of the still may, of course, influence this ratio; but Pacottet and Guittonneau³⁷ set 20 per cent as the maximum limit for beverage brandies to be distilled in pot stills. In steam-operated stills, where the danger of burning is less, a higher percentage may be permissible.

The most important fluctuations in composition of the product are due to the type and method of operation of the stills. Complete details of the influence of still operation on composition are given in the sections on still operation (p. 39 to 41). By removing some of the constituents which are more volatile than ethyl alcohol, such as volatile esters and aldehydes, and also some of the substances which are less volatile than ethyl alcohol, such as propyl, isobutyl, and other higher alcohols, the still operator is able to influence the quality of the product.

Other operational factors which are said to influence the composition of the product are the method of heating; the composition of the metal in the still; and, of course, the construction of the still—for example, pot or continuous still.

Furfural is present in the original wine and is the chief substance formed during distillation, but in most cases the amounts formed are small, 1 to 2 mg per liter of 100 per cent alcohol. Marc brandies, however, are very high in furfural, owing to the increased rate of formation of furfural in a pot still containing pomace. Direct-fired pot stills yield the greatest amounts of furfural.

Barbet³⁸ has recently proposed leaching the lees with low-proof spirits and making the final distillation under vacuum in order to add vitamins to the product. He also proposes use of high-vitamin fortifying brandy in the production of dessert wines. The practicability of the scheme has not been demonstrated. Addition of certain vitamins, particularly B₁, to spirits as an aid in the control of alcoholism has also been proposed in this country, as previously mentioned (p. 9).

Substances Added after Distillation.—Water and caramel are the two substances commonly added to California beverage brandies. Fortifying brandies are not ordinarily diluted or ameliorated before use.

³⁷ Pacottet, P., and L. Guittonneau. *Eaux-de-vie et vinaigres*. 480 p. Librairie J.-B. Baillière et Fils, Paris, France. 1926.

³⁸ Barbet, É. *Le vin, l'eau-de-vie et les vitamines*. *Mémoires de la Société des Ingénieurs Civils de France* 90(4):498-514. 1937.

The purpose of the addition of water to beverage brandies is to reduce the proof from that at which the brandy is distilled to about 102°; the proof may be reduced still further, to about 85°, after aging. In some cases, at least, cognac is not diluted until the time of marketing; the aging therefore is started at about 140° proof, and the alcohol content gradually decreases with time.

The purpose of caramel is to give the spirits color and a slight sweet taste. About 3 to 5 fluid ounces are added to a 50-gallon barrel. Cane sugar is sometimes added to cognacs, but the amount is limited to 2 per cent.

Addition of substances to beverage brandies which artificially enhance their characteristics constitutes, in most countries of the world, including France and the United States, a fraudulent practice unless due acknowledgment is made on the label (in this country the addition of the word "imitation"). In the United States, however, small amounts of harmless coloring, flavoring, and blending material, such as fruit juices, may be added under government supervision. Greek brandies sometimes have a slight resinous taste, but this may be derived from the original wine rather than from direct addition, or from improperly cleaned wooden containers which have previously contained the typical resinated Greek wines.

Changes during Aging.—Numerous changes take place in beverage brandies during aging. These changes result from substances derived from the wood and from chemical changes, mainly oxidation, alcoholysis, and esterification. Fortifying brandies are, however, seldom aged. (See p. 58.)

Substances derived from wood include acids, coloring substances, tannin, and woody tastes. Reif³⁹ has found vanillin in very small amounts (0.0 to 0.2 mg per liter of spirits); the amounts found were roughly proportional to the brown color of the spirits. Small increases in furfural occur during aging. This increase is much greater for spirits stored in charred barrels. The marked increase in solids is due to the substances dissolved from the wood. The increase in total acid is also in part due to substances derived from the wood.

California brandies are usually aged in new oak cooperage. French brandies are regularly aged in used cooperage. The rate of darkening is slower with used cooperage, and certainly the pickup of woody taste will be markedly reduced; less tannin extract will probably be absorbed in an equal period of time. The temperature of storage is generally higher for California brandies.

³⁹ Reif, G. Über das Vorkommen von Vanillin im Weindestillat, Weinbrand und Weinbrandverschnitt. Zeitschrift für Untersuchung der Lebensmittel 54:90-101. 1927.

The losses in weight during aging are considerable, as the following figures indicate:

| Type of barrel and period aged | Loss, in gallons per barrel |
|---------------------------------------|-----------------------------|
| Re-used, 4 years..... | 6.43 |
| New, plain, white oak: | |
| 1½ years | 4.47 |
| 2 years | 4.44 |
| 4 years, sample 1..... | 8.44 |
| 4 years, sample 2..... | 8.36 |
| New, charred, white oak, 2 years..... | 4.14 |
| Paraffined, 2 years..... | 1.1 |

Owing to differential loss of water, small increases in proof occur during the normal aging of California commercial brandies at low humidities if the initial proof is about 100°. This is apparently not true, however, for brandies whose initial proof at barreling is much higher than 100°, or those which are stored for very long periods of time, particularly at higher relative humidities. Jackson⁴⁰ reports, for example, that very old cognacs, initially over 100° proof, are only 76° to 78° proof. The type of cooperage, the conditions and length of storage, and the possibility of dilution during aging are also factors.

In a study of the changes occurring during the aging of cognacs extending from 1910 to 1934, Bellet⁴¹ found oxidation changes to be phenomena of less importance than had previously been assumed. The total esters remained essentially constant, but the more volatile esters rose very slowly at the expense of the less volatile esters of the higher alcohols. The consequent liberation of higher alcohols is called "alcoholysis." Some of the higher alcohols released are converted to aldehydes. The secondary importance of oxidation and the relatively greater importance of the slow process of alcoholysis indicate, according to Bellet, why rapid oxidation of new brandies fails to give satisfactory and practical results.

Valaer⁴² found that during the early aging of brandy of about 100° proof there was an increase in alcohol and in total and volatile acids, a very slight increase in esters and higher alcohols, and a definite increase in solids. Typical data are shown in table 6. Increases in proof, acids, esters, higher alcohols, solids, and other ingredients are slower in the plain barrels used for brandy and the increases are more uni-

⁴⁰ Jackson, G. H. The medicinal value of French brandy. 315 p. Thérien Frères, Montreal, Canada. 1928.

⁴¹ Bellet, E. M. Quelques observations sur les transformations chimiques qui accompagnent le vieillissement des eaux-de-vie. Société Chimique de France Bul. 4 (série 5):713-15. 1937.

⁴² Valaer, Peter. Chemical changes in brandy storage. The Wine Review 7(10): 14-16. 1939.

formly distributed over the whole period of aging, than in the new charred cooperage ordinarily used for whiskey and rum, where the greatest changes occur during the first six months.

The changes taking place in stored brandy are similar to those in whiskey and in rum. New charred barrels cause the most extensive changes; new plain barrels, re-used barrels, and paraffined barrels are next in order of changes. Even in the paraffined barrels, a gradual change takes place in the brandy, resulting in a slow increase in congeneries,

TABLE 6
CHANGES TAKING PLACE DURING STORAGE OF CALIFORNIA BRANDIES*

| Age | Proof | pH | Total acids | Volatile acids | Esters | Higher alcohols | Solids | Ash | Aldehydes | Furfural | Color in $\frac{1}{2}$ -in. cell† |
|----------------------|----------------|-----------|------------------------|------------------------|------------------------|------------------------|------------------------|------------------------|------------------------|------------------------|-----------------------------------|
| <i>years</i> | <i>degrees</i> | <i>pH</i> | <i>grams per 100 l</i> | <i>grams per 100 l</i> | <i>grams per 100 l</i> | <i>grams per 100 l</i> | <i>grams per 100 l</i> | <i>grams per 100 l</i> | <i>grams per 100 l</i> | <i>grams per 100 l</i> | |
| 0..... | 100.0 | 6.78 | 3.6 | 3.6 | 24.6 | 75.2 | 58 | 9 | 2.0 | 0.00 | 5.0 |
| $\frac{1}{2}$ | 100.0 | 5.50 | 9.6 | 7.2 | 25.8 | 76.6 | 68 | 6 | 3.0 | .00 | 4.5 |
| 1..... | 100.0 | 5.12 | 14.4 | 13.2 | 26.0 | 77.2 | 70 | 6 | 3.2 | .14 | 5.0 |
| $1\frac{1}{2}$ | 100.6 | 5.00 | 16.8 | 15.6 | 26.3 | 77.4 | 79 | 7 | 3.4 | .16 | 5.0 |
| 2..... | 101.5 | 5.00 | 19.2 | 18.8 | 26.3 | 79.2 | 72 | 6 | 3.4 | .16 | 5.0 |
| $2\frac{1}{2}$ | 101.5 | 4.92 | 21.6 | 20.4 | 26.4 | 82.2 | 82 | 9 | 3.0 | .20 | 5.5 |
| 3..... | 101.6 | 4.85 | 24.0 | 21.6 | 29.0 | 82.7 | 84 | 6 | 3.3 | .30 | 5.0 |
| $3\frac{1}{2}$ | 102.3 | 4.83 | 26.0 | 22.8 | 30.4 | 83.5 | 88 | 17 | 3.5 | .40 | 5.5 |
| 4..... | 102.8 | 4.82 | 27.6 | 25.3 | 31.7 | 84.5 | 94 | 4 | 3.7 | 0.40 | 6.0 |

* Grape brandy made from wine distilled in a copper continuous still using beer column only and stored in re-used plain barrels. Brandy was colored with caramel at time of filling. Samples gave positive tests for methyl alcohol (0.05 per cent). Ash was white and contained some iron. The barrels were set aside in United States bonded warehouses for experimental purposes. Proof of distillation, 168°-178°. Exclusive of samples taken, 6.43 wine gallons of brandy was lost from the barrel in four years.

† In a Lovibond tintometer apparatus.

Source of data:

Valaer, Peter. Brandy. Industrial and Engineering Chemistry, industrial edition 31:339-53. 1939

a slight increase in color, and a small loss of volume. There is more furfural in the brandy aged in the charred barrels than in that aged in plain re-used barrels.

One of the most apparent changes is in the ratio of esters to total acids. In the new brandy the esters are usually present in larger amounts, but during the aging in barrels, the ester increase is much less than the acid increase; after a year or so in storage, these values are not so far apart. Generally, however, the esters still exceed the acids. The analysis of changes in volatile and total acids in cognac are shown in table 7. There is some indication that the volatile acids decrease in relation to the total acids.

There is no apparent change in the methyl alcohol content during aging. In connection with changes in brandies during aging, the assertion that a substance actually increases in absolute amount must be

made with caution, for the apparent increase may merely reflect the increase in proportion of total congenics which takes place as a result of the preferential loss of alcohol during storage.

Substances Removed or Added by Treatments during Aging.—Rapid aging of brandies by addition of oak chips is relatively uncommon, either in California or elsewhere. The color and wood flavor are markedly increased by this treatment, especially if the barrels have been warmed.

Other treatments may be considered by the government as rectification and are usually not desirable.

TABLE 7
CHANGES IN TOTAL AND VOLATILE ACID IN COGNAC

| Age | Acid, as acetic, per 100 cc of absolute alcohol | | | Volatile as per cent of total acid |
|--------------|---|------------|---------------|------------------------------------|
| | Total acid | Fixed acid | Volatile acid | |
| <i>years</i> | <i>mg</i> | <i>mg</i> | <i>mg</i> | <i>per cent</i> |
| <1..... | 8.9- 36.4 | 1.2- 7.1 | 6.8- 29.5 | 71-87 |
| 1-3..... | 22.6- 84.0 | 3.5- 18.8 | 19.8- 65.1 | 72-90 |
| 5-11..... | 32.3-100.3 | 5.6- 36.2 | 21.1- 72.3 | 60-85 |
| 16-21..... | 70.3-142.0 | 17.7- 58.2 | 47.6-104.2 | 66-85 |
| 22-39..... | 110.9-300.0 | 29.6-126.8 | 81.3-173.2 | 57-73 |
| 44-80..... | 126.9-393.0 | 51.2-174.2 | 60.0-265.5 | 47-68 |

Source of data:

Rocques, W., and F. Lusson. Sur l'acidité fixe et l'acidité volatile des eaux-de-vie. Annales de Chimie Analytique Appliquée à l'Industrie, à l'Agriculture, à la Pharmacie et à la Biologie. 2:308-11. 1897.

It is occasionally necessary to remove off-odors and tastes from beverage brandies. Caffre⁴³ reports that about 1 pound of activated charcoal to 25 gallons of brandy eliminated a persistent bad taste. The furfural, esters, aldehydes, and acidity were also greatly reduced by the treatment; and the brandy lost most of its organoleptic properties. There was no change in the higher alcohols.

Excessive woody tastes are sometimes removed by the use of gelatin, about 28 grams to 280 liters (1 ounce to 74 gallons). Removal of iron and other metallic impurities has also been reported; but, aside from the legal problems involved, it is questionable if the value of the spirits after such treatment warrants the treatment, especially since it costs only a little less than redistillation.

⁴³ Caffre, J. Emploi du charbon activé dans la désodorisation des eaux-de-vie. Annales des Falsifications et des Fraudes 21:604. 1928.

CLASSIFICATION OF BRANDIES^a

Numerous types of grape brandy are produced in various parts of the world. The distinctiveness of the brandies depends on the combined influence of all the factors already discussed. Differences in the method of distillation are considered to be the most important, especially where local modifications in the process of distillation have been made which are closely integrated to the particular character of the raw material so as to produce a distinctive product. It is difficult, therefore, to establish for any particular brandy the relative importance of the raw material, method of handling, method of distillation, and nature of the aging process. The best method of classification, therefore, seems to be one which takes into account both the geographical origin of the spirits and the type of distillation process.

The United States Treasury Department defines several classes of brandies as follows:

'Brandy' is a distillate, or a mixture of distillates, obtained solely from the fermented juice, mash or wine of fruit, or from the residue thereof, distilled at less than 190° proof in such manner as to possess the taste, aroma and characteristics generally attributed to the product, and bottled at not less than 80° proof; and shall also include such distillates, aged for a period of not less than fifty years, and bottled at not less than 72° proof, in cases where the reduction in proof below 80° is due solely to losses resulting from natural causes during the period of aging. Brandy, or mixtures thereof, not conforming to any of the following standards shall be designated as 'brandy,' and such designation shall be qualified by a truthful and adequate statement of composition in direct conjunction therewith.

(a) 'Fruit brandy' is brandy distilled solely from the juice or mash of whole, sound, ripe fruit, or from standard grape, citrus, or other fruit wine, with or without the addition of not more than 20% by volume (calculated prior to the addition of water to facilitate distillation) of the lees of such wine, and shall include mixtures of such brandy with not more than 20% (calculated on a proof basis) of lees brandy. Fruit brandy, derived exclusively from grapes, shall be designated as 'grape brandy' or 'brandy.' Fruit brandy, other than grape brandy, derived exclusively from one variety of fruit, shall be designated by the word 'brandy' qualified by the name of such fruit (e.g., 'peach brandy,' 'apple brandy,' 'orange brandy') except that 'apple brandy' may be designated 'applejack.' Fruit brandy derived from more than one variety of fruit shall be designated as 'fruit brandy,' qualified by a truthful and adequate statement of composition (e.g., 'fruit brandy—a blend of 90% grape brandy and 10% blackberry brandy').

(b) 'Cognac' or 'Cognac (grape) brandy' is grape brandy distilled in the Cognac region of France, which is entitled to be so designated by the laws and regulations of the French government.

(c) 'Dried fruit brandy' is brandy that conforms to the standard for fruit brandy except that it has been derived from sound, dried fruit, or from the standard wine of

^a General references on this subject in addition to those given in specific footnotes in the section are listed on p. 74.

such fruit. Brandy derived from raisins, or from raisin wine, shall be designated as 'raisin brandy.' Other brandies defined in this paragraph shall be designated in the same manner as fruit brandy from the corresponding variety or varieties of fruit except that the name of the fruit shall be qualified by the word 'dried.'

(d) 'Lees brandy' is brandy distilled from the lees of standard grape, citrus, or other fruit wine, and shall be designated as 'lees brandy,' qualified by the name of the fruit from which such lees are derived.

(e) 'Pomace brandy,' or 'mare brandy,' is brandy distilled from the skin and pulp of sound, ripe grapes, citrus or other fruit, after the withdrawal of the juice or wine therefrom, and shall be designated as 'pomace brandy,' or 'mare brandy,' qualified by the name of the fruit from which derived. Grape pomace brandy may be designated as "grappa" or 'grappa brandy.'

(f) 'Neutral brandy' is any brandy distilled on or after July 1, 1941 at more than 170° proof. Brandy so distilled shall be designated in the same manner as if distilled at a lower proof, except that the designation shall be qualified by the word 'neutral' in the same size and kind of type, e.g., 'neutral brandy,' 'neutral grape lees brandy,' or 'neutral grape pomace brandy.'

(g) 'Substandard' brandy shall bear as a part of its designation the word 'substandard,' and shall include—

(1) Any brandy distilled at not more than 170° proof from juice, mash or wine having a volatile acidity in excess of that prescribed for standard wine, and any brandy distilled at more than 170° proof from juice, mash or wine having a volatile acidity, calculated as acetic acid and exclusive of sulphur dioxide, in excess of 0.20 gram per 100 cubic centimeters (20° C.); measurements of volatile acidity under this subparagraph shall be calculated exclusive of water added to facilitate distillation.

(2) Any brandy which has been distilled from unsound, mouldy, diseased or decomposed juice, mash or wine, or which shows in the finished product any taste, aroma or characteristic associated with products distilled from such juice, mash or wine.⁴⁵

CALIFORNIA

The great majority of California brandies have been produced from wine in continuous column stills.⁴⁶

A number of important pot-still brandies were produced before Prohibition. General Vallejo's still in 1835 in Sonoma County is noted by Scott.⁴⁷ Sutter's at Sacramento⁴⁸ preceded the gold rush. Colonel Naglee's⁴⁹ followed at a later date in Santa Clara, and he achieved some fame with his brandies, as did other early distillers in various sections. But the

⁴⁵ United States Treasury Department. Revised standards of identity for brandy. Treasury Decision 5050:1-3. 1941. (Effective July 1, 1941.)

⁴⁶ The continuous still, sometimes called a "patent" or "Coffey" still, is described on p. 42, and the pot still on p. 41.

⁴⁷ Scott, W. Brandy distillation, historical and statistical. In: Appendix A to the Biennial Report of the Board of State Viticultural Commissioners. 125 p. California State Printing Office, Sacramento, Calif. 1892.

⁴⁸ Jones, L. California's important brandy industry. Wines and Vines 17(10):3, 24. 1936.

⁴⁹ Bleasdale, J. I. The report of a jury of experts on brandy made by Gen. H. M. Naglee. Mechanics Institute of San Francisco Annual Rept. 13:1-114. 1879.

ordinary and regular commercial brandies appear to have been made with continuous column stills, particularly in the period after 1900. Even the well-known brandy which Governor Stanford made at Vina is said to have been mainly a continuous-still product.⁵⁰

Post-Repeal.—Two types of beverage brandies are now being produced in this state: grape brandy and muscat brandy. Since Repeal, practically all of this brandy has been produced in continuous column stills. Interest in pot-still brandies has not been great. The cost of production and the deficiency of suitable dry white wine for distilling material have also been factors involved in the widespread use of continuous stills.

California beverage brandies are sold at from 85° to 100° proof after having been aged in new oak cooperage. Under these conditions, a considerable amount of woody flavor is found in some brandies. The common French cognac sold in this country has only 80° to 88° proof (see table 9). This is a desirable practice. Undiluted brandy of 100° proof is usually not consumed with pleasure. And after dilution to high-ball strength with soda or water, the better-quality brandies do not compare so favorably with the poor-quality products, or at least, the lesser-quality brandies do not seem so bad. Henderson⁵¹ has also shown that spirits of high proof are much more conducive to alcoholism than those of lower proof and recommends lowering the minimum limits to 70° or even to 60° proof. It is true, of course, that brandy of 100° proof has at present a certain commercial advantage in the minds of many people. This is because government regulations require "bottled-in-bond" brandy to have this proof, and "bottled-in-bond" does serve as a measure of quality in this country.

FRANCE

The two most important beverage brandies produced from wine in France are cognac and armagnac. The Charente district to which cognac production is restricted lies north of Bordeaux; the Armagnac district is south of Bordeaux. Both types were originally and remain essentially pot-still brandies.⁵² Wine of low alcohol and high total acid is mainly

⁵⁰ Twight, E. H. Personal communication.

⁵¹ Henderson, Y. The high proof liquor as a factor in the production of alcoholism. *Quarterly Journal of Studies on Alcohol* 1:1-12. 1940.

See also citation in footnote 7, p. 5.

⁵² Pacottet, P., and L. Guittonneau. (Cited in footnote 37, p. 19.)

Cruss, W. V. Notes on Cognac brandy. *Fruit Products Journal* 19:301-3. 1940.

Peaz, Antonio Dal. Cognac distillation and manufacture. Translated by William G. Spencer for the Board of State Viticultural Commissioners. Appendix A to the Biennial Report of the Board of State Viticultural Commissioners for 1891-92. Part II. p. 72-125. State Printing Office, Sacramento, Calif. 1892.

used for distillation. The composition of some typical wines is given in table 8.

The lees are distilled with the wine. With cognac the first distillation in the pot still is made at about 54° proof and the second at only 140° to 150° proof. (See p. 41.) With armagnac only one distillation is made. The spirits are aged in used oak cooperage of about the same size as our barrels.

Because of its fame, the term "cognac" has been widely used in various countries for beverage brandies. The use of direct-fired pot stills, the limestone soils, high-acid grapes, low-alcohol wines, the accurate control

TABLE 8
COMPOSITION OF CERTAIN FRENCH WINES TO BE USED
FOR DISTILLATION

| Wine | Number of samples | Alcohol | Extract | Total acid as tartaric* |
|-----------------|-------------------|------------------------|------------------------|-------------------------|
| | <i>number</i> | <i>volume per cent</i> | <i>grams per 100cc</i> | <i>grams per 100cc</i> |
| Charente..... | 1 | 8.2 | 1.85 | 0.76 |
| Fins bois..... | 7 | 8.1 | 1.78 | — |
| Bons bois..... | 3 | 8.4 | 1.78 | — |
| Bois..... | 8 | 8.1 | 1.71 | — |
| Champagne†..... | 2 | 7.7 | 1.55 | 0.80 |
| Armagnac..... | 9 | 9.6 | 1.52 | — |

* Acids low, probably because of acid-reducing bacteria.

† Grande and Petite Champagne in the Cognac district.

Source of data:

Jacquet, L. La fabrication des eaux-de-vie. 227 p. G. Masson, Paris, France.

of the distillation by organoleptic examination, and the aging program all appear to be factors involved in the distinctiveness of the French product. Armagnac brandies are usually considered to be softer and less fine than cognacs.

Cognac decreases in proof with time, and it is reduced further, if necessary, to about 80° to 90° at the time of selling. (See table 9.)

GREECE

The large-scale distillation industry in Greece dates to the period of phylloxera in France when dried grapes of the Corinth variety could not be exported to France. The dried grapes were watered and fermented and the raisin wine produced was distilled. (See Pacottet and Guittonneau, cited in footnote 37, p. 19.) The industry, however, antedates this, for Thudichum and Dupré⁵³ report brandy production in Greece prior to 1872.

⁵³ Thudichum, J. L. W., and A. Dupré. A treatise on the origin, nature, and varieties of wine. 760 p. Macmillan and Co., Ltd., London, England. 1872.

TABLE 9
 COMPOSITION OF VARIOUS TYPES OF BRANDIES

| Distilling material, type of still, and origin | Proof | Solids | Total acid as acetic | Volatile acid as acetic | pH | Esters as ethyl acetate | Higher alcohols | Aldehyde | Furfural | Methyl alcohol |
|---|---------|----------|----------------------|-------------------------|------|-------------------------|-----------------|----------|----------|----------------|
| | degrees | mg per l | mg per l | mg per l | pH | mg per l | mg per l | mg per l | mg per l | mg per l |
| From wine, continuous still: | | | | | | | | | | |
| California,* | | | | | | | | | | |
| 114 samples: ^a | | | | | | | | | | |
| Minimum... | 99.4† | 48 | 4.8 | 4.8 | 3.85 | 20.2 | 14.1 | 1.4 | T | T |
| Maximum... | 125.0† | 210 | 101.0 | 88.0 | 5.67 | 180.4 | 250.0 | 24.0 | 5.0 | 0.188 |
| Average... | 103.7† | 130 | 54.8 | 42.9 | 4.42 | 68.9 | 90.7 | 10.7 | 1.7 | .048 |
| Languedoc, 11 samples: ^b | | | | | | | | | | |
| Minimum... | 97.4 | — | 4.6† | — | — | 32.5† | 55.0† | 7.0† | 0.0 | — |
| Maximum... | 138.6 | — | 96.5† | — | — | 145.2† | 210.0† | 30.0† | T | — |
| Average... | 121.4 | — | 32.9† | — | — | 85.5† | 114.1† | 13.8† | — | — |
| From wine, pot still: | | | | | | | | | | |
| Cognac, 43 samples: ^{a, d} | | | | | | | | | | |
| Minimum... | 63.6§ | 624 | 26.4 | 21.0 | 3.76 | 36.1 | 89.8 | 6.3 | 1.0 | .011 |
| Maximum... | 88.2§ | 1,368 | 110.4 | 72.0 | 4.98 | 58.1 | 383.1 | 14.0 | 3.0 | .027 |
| Average... | 84.6§ | 975 | 51.5 | 34.2 | 4.14 | 44.3 | 133.8 | 8.6 | 1.3 | .017 |
| Armagnac, 5 samples: ^{a, d} | | | | | | | | | | |
| Minimum... | 84.4 | 886 | 62.4 | 43.2 | 3.70 | 49.3 | 91.5 | 8.7 | 0.6 | .021 |
| Maximum... | 129.0 | 962 | 67.2 | 45.6 | 3.88 | 59.8 | 148.4 | 9.5 | 1.0 | .031 |
| Average... | 96.6 | 929 | 64.0 | 43.2? | 3.77 | 52.8 | 109.6 | 9.1 | 0.7 | .026 |
| Greek, [†] 35 samples: ^{a, e} | | | | | | | | | | |
| Minimum... | 80.2 | 198 | 21.6 | 6.0 | 3.28 | 7.9 | 10.6 | 4.0 | 0.0 | T |
| Maximum... | 94.0 | 4,130 | 103.2 | 70.8 | 5.77 | 79.2 | 121.4 | 23.6 | 2.4 | .050 |
| Average... | 87.4 | 1,772 | 51.9 | 33.8 | 4.41 | 40.5 | 58.0 | 13.8 | 1.0 | — |
| From wine, vacuum still: | | | | | | | | | | |
| Languedoc, 9 samples: ^b | | | | | | | | | | |
| Minimum... | 83.2 | — | 4.3† | — | — | 22.8† | 44.5† | 7.3† | 0.0 | — |
| Maximum... | 146.2 | — | 48.1† | — | — | 42.3† | 88.5† | 9.6† | T | — |
| Average... | 108.2 | — | 16.1† | — | — | 34.8† | 61.3† | 7.9† | — | — |
| From marc, pot still: | | | | | | | | | | |
| Burgundy, 8 samples: ^d | | | | | | | | | | |
| Minimum... | 83.0 | — | — | — | — | — | — | — | — | — |
| Maximum... | 102.8 | — | — | — | — | — | — | — | — | — |
| Average... | 97.2 | — | — | — | — | — | 127.3 | — | — | — |
| Languedoc 7 samples: ^b | | | | | | | | | | |
| Minimum... | 103.8 | — | 12.8† | — | — | 49.4† | 125.0† | 16.0† | 0.0† | — |
| Maximum... | 137.0 | — | 120.0† | — | — | 344.0† | 200.0† | 288.0† | 0.9† | — |
| Average... | 120.4 | — | 33.2† | — | — | 111.4† | 153.1† | 157.4† | 0.7† | — |
| From wash, continuous still: | | | | | | | | | | |
| Languedoc, 6 samples: ^b | | | | | | | | | | |
| Minimum... | 129.0 | — | 2.3† | — | — | 20.3† | 85.0† | 11.7† | 0.0† | — |
| Maximum... | 137.4 | — | 19.8† | — | — | 140.8† | 125.0† | 76.0† | T | — |
| Average... | 133.4 | — | 10.6† | — | — | 59.3† | 105.7† | 31.8† | — | — |

* Not all were distilled in continuous stills but most samples were. Several abnormal samples not included in averages. Many of these were bulk brandy samples hence the proof is higher than that of many present-day bottled California brandies.

† Apparent proof. ‡ Calculated to mg per l liter of 100° proof spirits.

§ True proof. Upper limit of Mathieu samples omitted; Mathieu analysis only for alcohol and higher alcohols.

¶ About two thirds of the samples were distilled in pot stills, the others in continuous stills. Data from Miermeister on proof, solids, and furfural only.

|| True proof.

Sources of data:

^a Valaer, P. Brandy. Industrial and Engineering Chemistry, industrial edition 31:339-53. 1939.

^b Flanzy, M., and M. Lamazou-Betbeter. Sur la composition des eaux-de-vie de la région languedocienne. Annales de Technologie Agricole 1:106-19. 1938.

^c Miermeister, A. Über Zusammensetzung und Herstellung griechische Weinbrände. Chemiker-Zeitung 56:371-72. 1932.

^d Mathieu, L. Les grandes eaux-de-vie françaises et la nouvelle loi belge. Revue de Viticulture 19:186-90, 216-19, 273-77. 1903.

The best modern description of the Greek brandy is that of Valaer (cited in footnote 20, p. 10). He reports that a young wine of rather high alcohol and low acidity is used for the distillation in a simple or modified pot still or, in the case of about one third of the Greek brandies, in column stills. The distillation may take place in two steps, as with cognac, or in a single distillation, as with armagnac. The proof of distillation is from 134° to 156°. The brandy is aged in fairly large oak containers.

The Greek brandies are higher in caramel than others, and according to table 9 have a lower acid and ester content and a somewhat higher aldehyde content. Valaer also reports that some samples were flavored with imitation brandy flavor as well as with anise and wine.

OTHER COUNTRIES

Spain.—Only a few Spanish brandies (*aguardientes*) are imported into the United States, but the Spanish distilling industry, in normal times, is a large one. González Gordon⁵⁴ reports that from 1909 to 1934 the average yearly shipment of beverage brandy from the Jerez de la Frontera district alone amounted to nearly one million gallons. The wines of the district are used, and the majority of the stills are of the pot type.

Portugal.—Only a few Portuguese brandies are found on the market. The best are produced in pot stills, although continuous stills are also used, from wines containing a fairly high percentage of alcohol. They are aged in oak for several years.

Italy.—There is a very large brandy industry in Italy. Most of it, however, is for the production of fortifying brandy for the fortification of Marsalas, vermouths, and related wines. In recent years much progress has been made in Italy in the recovery of alcohol from grape pomace—and even in the extraction of oil from the seeds.

Peru.—The production of *pisco* brandy in Peru has achieved considerable fame. According to Pacottet and Guittonneau, several brandies are produced but the most important is a muscat brandy which is commonly called *pisco*. *Pisco* brandy achieved some fame in California during the period just after the gold rush in the making of “pisco punch.”

POMACE BRANDIES

The pomace, after fermentation and pressing, still contains considerable alcohol. In several districts this pomace is distilled in special pot stills and the recovered alcohol reduced and aged. The *eaux-de-vie de marc* of France is distilled from relatively fresh pomace and is then aged for a number of years in oak.

⁵⁴ González Gordon, M. M^a. Jerez-Xerex-“Scherris.” 405 p. (See especially p. 363-96.) Imprenta A. Padura, Jerez de la Frontera, Spain. 1935.

The grappa brandy made in California is a grape-pomace product which, however, is usually made from pomace which has been stored. During storage the pomace undergoes some anaerobic decomposition, and some highly aromatic substances are produced in the interior of the pomace pile. The acetified external layer is removed before distillation. The remaining pomace is usually distilled in large pot stills and the product stored in paraffined barrels. It reaches the market in a nearly water-white condition. Grappa has a very pungent aroma, which is pleasing to some consumers and less pleasing to others.

FORTIFYING BRANDIES

Fortifying brandies are made from wine, wash from grape pomace, or from the pomace itself. Because of their very high proof of distillation, practically all of the higher alcohols, and aldehydes and esters as well, may be removed. In California fortifying brandy is commonly produced at 180° to 189° proof. (See p. 51.)

Continuous stills are commonly used for producing fortifying brandy since pot stills are too slow and expensive to operate. Modified continuous column stills which handle disintegrated or macerated pomace are also used. Greater recovery of alcohol from the pomace is claimed for these types of stills. When properly constructed and operated, they are a convenience in the handling of the pomace because the pomace is removed from the still in a liquid mass and may be disposed of as distillery slop.

Fortifying brandy should usually be neutral in reaction and nearly free of higher alcohols and undesirable flavors or odors. For certain fortification purposes, however, lower-proof material may occasionally prove useful. (See Bulletin 651, p. 57 and 78.)

The chief use of high-proof spirits is for fortification purposes, although some is diverted to other uses, particularly in France, for mixing with gasoline and for use in liqueurs.

PRINCIPLES OF BRANDY MAKING⁵⁵

PRODUCTION OF WINE

Securing desirable varieties of grapes for producing the most suitable wines to distill in making brandies is very important. The best brandies are produced from wines which are particularly suited to brandy production. The ideal wines seem to be those of moderate alcohol and above-average total acid. Low-acid wines are considered undesirable, particu-

⁵⁵ General references on this subject in addition to those given in specific footnotes in this section are listed on p. 74 to 76.

larly for use in pot stills. Red wines are not used for the best beverage brandies. (See p. 16.) Red wines and wines of low acidity are, however, not objectionable for the production of fortifying brandy that is highly rectified. The remarks to follow, although primarily directed towards the production of wine for distilling to produce beverage brandy, are likewise applicable, if less urgently so, to the production of fortifying brandy.

Varieties.—Varieties that produce wines satisfactory for nonmuscat commercial brandy include: Burger, Folle blanche, Green Hungarian, Saint Emilion, and, in addition, according to Wetmore,⁵⁶ West's White Prolific and Verdal. Neutral-flavored varieties, such as the Mission and Grenache, may also be used if they are picked sufficiently early and pressed off the skins before fermentation.

Muscat brandies are produced almost entirely from the Muscat of Alexandria in this state. This grape should be picked as soon as the muscat flavor develops and should not be allowed to remain on the vine until the acidity is reduced too low. Second-crop muscats also may be used. The use of muscat brandy to blend into the regular commercial brandies (or of muscat wine in the distilling material) in order to increase their character is common. The result is that only a few regular muscat brandies are produced, and many brandies have only a slight muscat character. Some restriction on the use of muscats in regular California brandies may therefore be desirable, if a uniform grape brandy is to be produced in this state. The muscat-flavored brandies should always be labeled as muscat brandy to avoid consumer confusion.

Except possibly for the Muscat of Alexandria, the other varieties for brandy production will produce more desirable wines for distillation when grown in the cooler districts. Analysis of varieties which are particularly useful are given in table 10. Of these the Folle blanche, when picked in good condition, is the best.

Harvesting and Fermentation.—The grapes should be picked while in good condition and before they begin to raisin. The presence of raisins is undesirable, not only because of the impairment of flavor but also because the complete extraction of all the sugar from them is difficult.

The grapes selected should be picked and transported to the winery with the same care and speed given to grapes to be made into white table wines. They should be crushed without delay and, except for musts to be used for muscat and fortifying brandies, the skins and free-run juice should be separated.

⁵⁶ Wetmore, C. J. Grapes suitable for fine brandy. p. 12-14. In: Brandy distillation. Appendix A to the Biennial Report of the Board of State Viticultural Commissioners for 1891-92. 125 p. California State Printing Office, Sacramento, Calif. 1892.

The use of very small amounts of sulfur dioxide (50 parts per million or less) and pure yeasts is considered advisable. The importance of pure yeasts is not known. Mixed cultures containing organisms which produce notable quantities of esters, particularly esters of the higher-molecular-weight homologues of acetic and butyric acids, may be desirable, but only if their activity can be controlled and does not also lead to the production of undesirable substances.

Excessive amounts of sulfur dioxide should not be used in wine to be distilled, for the presence of sulfur dioxide and compounds derived from it in the distillate gives the brandy an objectionable odor and taste.

TABLE 10
ANALYSES OF GRAPE VARIETIES COMMONLY USED FOR BRANDY PRODUCTION

| Variety | Region | Period tested | Average time of harvesting | Balling | Total acid as tartaric | pH | Balling | Desirability of the wine for distillation |
|----------------------------|--------------|---------------|----------------------------|---------|------------------------|------|---------|---|
| | | years | | degrees | grams per 100 cc | pH | ratio | |
| Burger..... | Davis..... | 2 | Sept. 21 | 22.0 | 0.68 | 3.59 | 32.2 | Fair |
| Burger..... | Santa Clara. | 3 | Oct. 8 | 19.3 | .68 | 3.23 | 28.7 | Fair |
| Folle blanche..... | Davis..... | 2 | Oct. 4 | 22.3 | .68 | 3.56 | 32.8 | Fair |
| Folle blanche..... | Santa Clara. | 6 | Oct. 3 | 19.9 | .83 | 2.99 | 24.0 | Very good |
| Green Hungarian. | Lodi..... | 3 | Sept. 30 | 19.7 | .45 | 3.55 | 43.8 | Poor |
| Green Hungarian. | Santa Clara. | 5 | Oct. 6 | 18.7 | .41 | 3.35 | 45.6 | Poor |
| Saint Emilion | Davis..... | 4 | Oct. 1 | 21.7 | .68 | 3.47 | 31.9 | Fair— |
| Saint Emilion | Santa Clara. | 2 | Oct. 18 | 24.7 | .76 | 3.16 | 32.5 | Fair— |
| West's White Prolific..... | Davis..... | 2 | Sept. 11 | 21.6 | 0.80 | 3.42 | 27.0 | Fair |

It also corrodes the plates and the inside surface of the still. (See also p. 63.)

The new wine may be distilled immediately after fermentation is completed. If distillation facilities are limited and the wine is to be stored for any length of time, it should be racked away from the lees before autolysis and reducing conditions occur in the sediment. Hydrogen sulfide and other bad-smelling substances are usually produced under such conditions. As previously indicated (p. 18), however, distilling the wine in contact with its own lees is considered to be of great value in the production of the best brandies in a pot still.

THEORY OF DISTILLATION

The separation of the constituents of a liquid mixture by partial vaporization of the mixture and separate recovery of vapor and residue is known as "fractional distillation." The more volatile constituents of the original mixture are obtained in increased concentration in the conden-

sate from the vapor, the less volatile in greater concentration in the liquid residue. The extent to which the constituents may be separated and the ease of such a separation depends on the nature of the mixture, and the manner of distillation.

Immiscible Liquids.—A mixture of immiscible liquids will boil at a temperature below that at which either of the component liquids would boil alone. If the liquids are to any extent mutually soluble, the individual vapor pressures are decreased and the mixture must be heated to a higher temperature to reach its boiling point, than for completely immiscible liquids. The boiling point is the temperature at which the sum of the vapor pressures of the individual components is equal to atmospheric pressure. The relative amounts of the components in the vapor as it passes into the condenser depends on the relative volatilities of the components as measured by their vapor pressures and is independent of the amount of each of the components present. It is due to this type of steam distillation that the fusel oil present in alcoholic solutions of low strength goes over in the first runnings and occurs in the foreshots, or "heads." This is the basis of the separation of ethyl alcohol from fusel oil in Guillaume's rectifying column, in which fusel oil is recovered at the top of the column together with the other impurities.

Miscible Liquids.—With two liquids that are mutually soluble, the vapor pressure of each is decreased by the presence of the other and therefore the sum of their vapor pressures is less than the sum of the vapor pressures of the two individual liquids. The boiling point of the solution will depend on the percentage composition and the nature of the components; it may be intermediate, below, or above that of the components. The composition of the vapor is profoundly influenced by the composition of the mixture, and so is the degree of separation by distillation. Ethyl alcohol and water form such a system.

Alcohol and Water Mixtures.—If a mixture of alcohol and water is boiled, the proportion of alcohol to water in the vapor is higher than it is in the liquid up to about 97.2 per cent. The relative distribution of alcohol in vapor and liquid varies according to the alcoholic strength of the latter. The results obtained by Sorel are shown in table 11. Later results by other workers are included for comparison.⁵⁷ The last column gives the ratio (k) between the percentage of alcohol in the vapor and that in the liquid. From the data presented, it is evident that by a process of repeated distillation and condensation it should be possible to raise the alcoholic strength of the distillate gradually until the product contains 97.6, or according to more recent determinations, 97.2 per cent of

⁵⁷ The differences in k values are due to failure of earlier workers to purify their alcohol sufficiently and to errors of technique in establishing the true boiling points.

TABLE 11
BOILING POINT AND COMPOSITION IN THE LIQUID AND VAPOR
OF ETHYL ALCOHOL AND WATER MIXTURES

| Per cent alcohol in liquid by weight | Boiling temperature | | | Per cent alcohol in vapor by weight | | | | Ratio (<i>k</i>) of alcohol content of vapor to that of the liquid | | | |
|--------------------------------------|---|------------------|--|-------------------------------------|------------------------|------------------|--|--|------------------------|------------------|--|
| | Sorel ^a and Groen-ing ^b | Gay ^c | Inter-natl. Critical Tables ^d | Sorel ^a | Groen-ing ^b | Gay ^c | Inter-natl. Critical Tables ^d | Sorel ^a | Groen-ing ^b | Gay ^c | Inter-natl. Critical Tables ^d |
| <i>per cent</i> | <i>°C</i> | <i>°C</i> | <i>°C</i> | <i>per cent</i> | <i>per cent</i> | <i>per cent</i> | <i>per cent</i> | <i>ratio</i> | <i>ratio</i> | <i>ratio</i> | <i>ratio</i> |
| 0..... | 100.0 | 100.0 | 100.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.00 | 0.00 | 0.00 | 0.00 |
| 1..... | 98.9 | 98.9 | 98.5 | 9.4 | 12.0 | 9.3 | 13.0 | 9.40 | 12.00 | 9.30 | 13.00 |
| 2..... | 97.8 | 97.9 | 97.1 | 18.0 | 26.0 | 17.6 | 23.6 | 9.00 | 13.00 | 8.80 | 11.80 |
| 3..... | 96.8 | 96.9 | 96.0 | 24.3 | 32.2 | 23.8 | 32.4 | 8.10 | 10.73 | 7.93 | 10.80 |
| 4..... | 95.9 | 95.9 | 94.8 | 30.0 | 36.5 | 28.9 | 39.4 | 7.50 | 9.13 | 7.23 | 9.85 |
| 5..... | 95.0 | 95.0 | 93.8 | 33.9 | 40.6 | 33.4 | 44.8 | 6.78 | 8.12 | 6.68 | 8.96 |
| 6..... | 94.1 | 94.2 | 92.9 | 37.6 | 43.8 | 37.3 | 49.4 | 6.27 | 7.30 | 6.22 | 8.23 |
| 7..... | 93.4 | 93.5 | 92.0 | 40.8 | 46.5 | 40.7 | 52.9 | 5.83 | 6.64 | 5.81 | 7.56 |
| 8..... | 92.6 | 92.7 | 91.3 | 43.5 | 49.2 | 43.9 | 55.9 | 5.44 | 6.15 | 5.49 | 6.99 |
| 9..... | 92.0 | 92.1 | 90.7 | 46.2 | 51.6 | 46.4 | 58.2 | 5.13 | 5.73 | 5.16 | 6.47 |
| 10..... | 91.3 | 91.4 | 90.1 | 49.3 | 53.6 | 48.8 | 60.4 | 4.93 | 5.36 | 4.88 | 6.04 |
| 11..... | 90.8 | 90.9 | 89.6 | 51.4 | 55.6 | 51.1 | 62.3 | 4.67 | 5.05 | 4.65 | 5.66 |
| 12..... | 90.2 | 90.2 | 89.1 | 53.6 | 57.6 | 53.5 | 64.0 | 4.47 | 4.80 | 4.46 | 5.33 |
| 13..... | 89.7 | 89.7 | 88.6 | 55.1 | 59.2 | 55.0 | 65.4 | 4.24 | 4.55 | 4.23 | 5.03 |
| 14..... | 89.2 | 89.3 | 88.1 | 56.5 | 60.7 | 56.3 | 66.9 | 4.04 | 4.34 | 4.02 | 4.78 |
| 15..... | 88.8 | 88.9 | 87.7 | 57.5 | 61.8 | 57.5 | 68.3 | 3.83 | 4.12 | 3.84 | 4.55 |
| 16..... | 88.4 | 88.4 | 87.3 | 58.3 | 63.2 | 58.0 | 69.5 | 3.64 | 3.95 | 3.62 | 4.35 |
| 18..... | 87.7 | 87.8 | 86.6 | 59.4 | 65.6 | 59.1 | 71.1 | 3.30 | 3.64 | 3.28 | 3.95 |
| 20..... | 87.0 | 87.1 | 85.9 | 60.2 | 67.7 | 60.2 | 72.6 | 3.01 | 3.39 | 3.01 | 3.65 |
| 22..... | 86.4 | 86.4 | 85.4 | 60.9 | 69.5 | 61.0 | 73.7 | 2.77 | 3.16 | 2.77 | 3.35 |
| 24..... | 85.9 | 85.9 | 84.8 | 61.6 | 71.0 | 61.7 | 74.7 | 2.57 | 2.96 | 2.57 | 3.11 |
| 26..... | 85.4 | 85.4 | 84.4 | 62.3 | 72.4 | 62.4 | 75.4 | 2.40 | 2.78 | 2.40 | 2.90 |
| 28..... | 85.0 | 85.0 | 84.0 | 62.9 | 74.0 | 63.0 | 76.2 | 2.25 | 2.64 | 2.25 | 2.72 |
| 30..... | 84.6 | 84.7 | 83.6 | 63.6 | 74.8 | 63.5 | 76.9 | 2.12 | 2.50 | 2.12 | 2.56 |
| 32..... | 84.3 | 84.3 | 83.2 | 64.3 | 75.7 | 64.3 | 77.5 | 2.01 | 2.37 | 2.01 | 2.42 |
| 34..... | 84.0 | 84.0 | 82.8 | 64.9 | 76.2 | 65.1 | 78.0 | 1.91 | 2.24 | 1.92 | 2.30 |
| 36..... | 83.7 | 83.6 | 82.5 | 65.6 | 77.3 | 65.8 | 78.5 | 1.82 | 1.14 | 1.83 | 2.18 |
| 38..... | 83.4 | 83.4 | 82.3 | 66.4 | 78.0 | 66.4 | 78.9 | 1.75 | 2.05 | 1.75 | 2.08 |
| 40..... | 83.2 | 83.1 | 82.1 | 67.2 | 78.8 | 67.0 | 79.3 | 1.68 | 1.96 | 1.68 | 1.98 |
| 42..... | 82.9 | 82.9 | 81.9 | 67.8 | 79.4 | 67.8 | 79.7 | 1.61 | 1.89 | 1.61 | 1.90 |
| 44..... | 82.6 | 82.6 | 81.7 | 68.4 | 80.0 | 68.4 | 80.0 | 1.55 | 1.82 | 1.55 | 1.82 |
| 46..... | 82.4 | 82.4 | 81.5 | 69.1 | 80.5 | 69.0 | 80.3 | 1.50 | 1.75 | 1.50 | 1.75 |
| 48..... | 82.2 | 82.2 | 81.3 | 69.9 | 81.1 | 69.7 | 80.6 | 1.46 | 1.69 | 1.45 | 1.68 |
| 50..... | 82.0 | 81.9 | 81.2 | 70.6 | 81.6 | 70.7 | 80.9 | 1.41 | 1.63 | 1.41 | 1.62 |
| 52..... | 81.8 | 81.8 | 81.0 | 71.5 | 82.0 | 71.2 | 81.3 | 1.37 | 1.58 | 1.37 | 1.56 |
| 54..... | 81.6 | 81.6 | 80.9 | 72.3 | 82.5 | 71.9 | 81.6 | 1.34 | 1.53 | 1.33 | 1.51 |
| 56..... | 81.4 | 81.4 | 80.7 | 73.1 | 83.0 | 72.7 | 81.9 | 1.31 | 1.48 | 1.30 | 1.46 |
| 58..... | 81.2 | 81.2 | 80.6 | 73.9 | 83.4 | 73.6 | 82.2 | 1.27 | 1.44 | 1.27 | 1.42 |
| 60..... | 81.0 | 81.0 | 80.4 | 74.8 | 83.8 | 74.6 | 82.5 | 1.25 | 1.40 | 1.24 | 1.37 |
| 62..... | 80.8 | 80.9 | 80.3 | 75.6 | 84.3 | 75.2 | 82.9 | 1.22 | 1.36 | 1.21 | 1.34 |
| 64..... | 80.7 | 80.7 | 80.1 | 76.4 | 84.7 | 76.1 | 83.2 | 1.19 | 1.32 | 1.19 | 1.30 |
| 66..... | 80.5 | 80.6 | 80.0 | 77.4 | 85.0 | 77.2 | 83.6 | 1.17 | 1.29 | 1.17 | 1.27 |

Sources of data:

^a Sorel, Ernest. *Cited by*: Monier-Williams, G. W. Power alcohol. 323 p. (Citation on p. 82-83.) Henry Frowde and Hodder and Stoughton, London, England. 1922.^b Groening, *Cited by*: Boullanger, E. Distillerie agricole et industrielle. Vol. II. 405 p. (Citation on p. 8-9.) Librairie J.-B. Baillière et Fils, Paris, France. 1925.^c Gay, M. Distillation and rectification. 661 p. (See especially p. 100.) Librairie J.-B. Baillière et Fils, Paris, France. 1935.^d Keys, D. B. Variation of boiling point with composition for liquid mixtures of volatile constituents. *In*: International critical tables. Vol. III. 444 p. (See especially p. 310.) McGraw-Hill Book Company, New York, N. Y. 1928.

TABLE 11—(Continued)

| Per cent alcohol in liquid by weight | Boiling temperature | | | Per cent alcohol in vapor by weight | | | | Ratio (<i>k</i>) of alcohol content of vapor to that of the liquid | | | |
|--------------------------------------|--|------------------|--|-------------------------------------|-----------------------|------------------|--|--|-----------------------|------------------|--|
| | Sorel ^a and Groening ^b | Gay ^c | Internat. Critical Tables ^d | Sorel ^a | Groening ^b | Gay ^c | Internat. Critical Tables ^d | Sorel ^a | Groening ^b | Gay ^c | Internat. Critical Tables ^d |
| <i>per cent</i> | <i>°C</i> | <i>°C</i> | <i>°C</i> | <i>per cent</i> | <i>per cent</i> | <i>per cent</i> | <i>per cent</i> | <i>ratio</i> | <i>ratio</i> | <i>ratio</i> | <i>ratio</i> |
| 68..... | 80.3 | 80.4 | 79.9 | 78.4 | 85.5 | 78.3 | 83.9 | 1.15 | 1.26 | 1.15 | 1.23 |
| 70..... | 80.2 | 80.2 | 79.7 | 79.4 | 85.9 | 79.4 | 84.2 | 1.13 | 1.23 | 1.13 | 1.20 |
| 72..... | 80.0 | 80.1 | 79.6 | 80.4 | 86.2 | 80.0 | 84.7 | 1.12 | 1.20 | 1.11 | 1.18 |
| 74..... | 79.8 | 79.9 | 79.4 | 81.7 | 86.6 | 80.9 | 85.3 | 1.10 | 1.17 | 1.09 | 1.15 |
| 76..... | 79.7 | 79.7 | 79.2 | 83.0 | 87.0 | 82.0 | 85.9 | 1.09 | 1.145 | 1.08 | 1.13 |
| 78..... | 79.6 | 79.6 | 79.0 | 83.9 | 87.4 | 82.9 | 86.6 | 1.075 | 1.145 | 1.07 | 1.11 |
| 80..... | 79.5 | 79.45 | 78.8 | 85.1 | 87.7 | 84.8 | 87.6 | 1.064 | 1.097 | 1.059 | 1.096 |
| 82..... | 79.3 | 79.2 | 78.6 | 86.1 | 88.1 | 85.4 | 88.4 | 1.051 | 1.075 | 1.040 | 1.078 |
| 84..... | 79.2 | 79.0 | 78.4 | 87.6 | 88.8 | 86.9 | 89.4 | 1.041 | 1.058 | 1.032 | 1.067 |
| 86..... | 79.1 | 78.8 | 78.35 | 88.8 | 89.2 | 88.3 | 90.2 | 1.032 | 1.038 | 1.026 | 1.049 |
| 88..... | 79.0 | 78.6 | 78.25 | 90.2 | 89.8 | 90.2 | 90.8 | 1.025 | 1.020 | 1.023 | 1.031 |
| 90..... | 78.9 | 78.4 | 78.21 | 92.0 | — | 91.0 | 91.0 | 1.021 | — | 1.012 | 1.011 |
| 91.2..... | — | — | 78.20 | — | — | — | 91.2 | — | — | — | 1.000 |
| 92..... | 78.75 | 78.25 | — | 93.4 | 92.5 | 92.8 | — | 1.015 | 1.005 | 1.010 | — |
| 93..... | 78.7 | 78.20 | — | 94.0 | — | 94.0 | — | 1.012 | — | 1.010 | — |
| 94..... | 78.7 | 78.18 | — | 94.8 | — | 94.8 | — | 1.008 | — | 1.008 | — |
| 95..... | 78.65 | 78.16 | — | 95.6 | — | 95.4 | — | 1.006 | — | 1.002 | — |
| 95.57..... | — | 78.15 | — | — | — | 95.57 | — | — | — | 1.000 | — |
| 96..... | 78.6 | — | — | 96.1 | — | — | — | 1.002 | — | — | — |
| 96.8..... | 78.6 | — | — | 96.8 | 96.8 | — | — | 1.000 | 1.000 | — | — |

Sources of data:

^a Sorel, Ernest. *Cited by*: Monier-Williams, G. W. Power alcohol. 323 p. (Citation on p. 82-83.) Henry Frowde and Hodder and Stoughton, London, England. 1922.^b Groening. *Cited by*: Boullanger, E. Distillerie agricole et industrielle. Vol. II. 405 p. (Citation on p. 8-9.) Librairie J.-B. Baillière et Fils, Paris, France. 1925.^c Gay, M. Distillation and rectification. 661 p. (See especially p. 100.) Librairie J.-B. Baillière et Fils, Paris, France. 1935.^d Keys, D. B. Variation of boiling point with composition for liquid mixtures of volatile constituents. *In*: International critical tables. Vol. III. 444 p. (See especially p. 310.) McGraw-Hill Book Company, New York, N. Y. 1928.

alcohol by volume (95.57 per cent by weight at 20° C; at 15.56° C, or 60° F, 97.2 per cent by volume corresponds to 95.62 per cent by weight). A mixture of this strength has a constant boiling point 172.7° F (78.15° C or about 0.4° F below that of pure alcohol) and distills unchanged in composition. It is therefore impossible to effect a further separation by distillation alone. Absolute alcohol can be prepared, however, by redistilling the spirit so obtained with the addition of a dehydrating agent such as calcium oxide or by azeotropic distillation.⁵⁸

Other Volatile Components.—Alcohol is not the only volatile constituent of the wine or distilling material. Volatile fermentation by-products such as acetaldehyde, esters, higher alcohols, and volatile acids are

⁵⁸ Dietrich, K. R. *Neuzeitliche Herstellungsverfahren für absoluten Alkohol*. Zeitschrift für Angewandte Chemie 43:40-44. 1930.Guinot, H. Industrial dehydration of alcohol. *International Sugar Journal* 32: 77-83. 1930.

TABLE 12
PROPERTIES OF SOME POSSIBLE CONSTITUENTS OF BRANDY

| Constituent | Specific gravity* | Boiling point | Boiling point | Solubility† | |
|-------------------------------|------------------------------------|---------------|---------------|---------------|---------------|
| | | | | In alcohol | In water |
| | | | | parts per 100 | parts per 100 |
| Alcohols: | | | | | |
| Methyl..... | 0.792 | 148.5 | 64.7 | ∞ ‡ | ∞ |
| Ethyl..... | 0.789 | 173.1 | 78.4 | ∞ | ∞ |
| Propyl..... | 0.804 | 208 (207) | 97.8 (97.19) | ∞ | ∞ |
| Isopropyl..... | 0.785 | 180.5 | 82.5 | ∞ | ∞ |
| Butyl..... | 0.810 | 242.6 | 117 | ∞ | 915°C |
| Isobutyl..... | 0.805 ¹⁷ / ₈ | 224.6 (227.1) | 107 (108.39) | ∞ | 1015°C |
| <i>n</i> -amyl..... | 0.817 ²⁰ / ₈ | 180 | 137.8 | ∞ | 2.722°C |
| Isoamyl..... | 0.812 | 275 | 130.5 | ∞ | 2.6722°C |
| Hexyl..... | 0.819 | 311.4 | 155.2§ | sol. | 0.5920°C |
| Heptyl..... | 0.819 | 348.8 | 176.3 | ∞ | 0.28100°C |
| 2, 3-Butylene, glycol..... | 1.048 ² / ₃ | 363.2 | 184 | sol. | ∞ |
| Aldehydes: | | | | | |
| Acetaldehyde..... | 0.783 ¹⁸ / ₄ | 68.4 | 20.2 | ∞ | ∞ |
| Furfural..... | 1.160 | 320.9 (323.1) | 160.5 (161.7) | ∞ | 9.113°C |
| Acids: | | | | | |
| Formic..... | 1.220 | 213.4 | 100.8 | ∞ | ∞ |
| Acetic..... | 1.049 | 244.6 | 118.1 | ∞ | ∞ |
| Lactic..... | 1.249 ¹⁵ / ₄ | 251.6 | 122 | ∞ | ∞ |
| Propionic..... | 0.992 | 286 | 141.1 | ∞ | ∞ |
| Butyric..... | 0.959 | 323.6 | 162 | ∞ | 5.621.1°C |
| Valeric..... | 0.942 | 368.6 | 187 | ∞ | 3.716°C |
| Esters: | | | | | |
| Ethyl formate..... | 0.906 | 129.2 | 54 | sol. | 11.825°C |
| Ethyl acetate..... | 0.901 | 170.8 | 77.1 | ∞ | 8.515°C |
| Ethyl propionate..... | 0.891 | 210.4 | 99.1 | ∞ | 2.420°C |
| Ethyl butyrate..... | 0.879 | 248.2 (250.3) | 120.1 (121.3) | sol. | 0.6825°C |
| Ethyl isovalerate..... | 0.866 | 275 | 135 | ∞ | 0.1720°C |
| Ethyl <i>n</i> -valerate..... | 0.877 ²⁰ / ₈ | 292.1 | 144.5§ | ∞ | 0.2425°C |
| Isoamyl acetate..... | 0.876 ¹⁵ / ₄ | 287.6 | 142§ | ∞ | 0.2515°C |
| Isoamyl butyrate..... | 0.866 ¹⁹ / ₈ | 354.2 | 179¶ | ∞ | 0.0550°C |
| Ethyl caproate..... | 0.873 | 332.1 | 166.7 | sol. | 0.001520°C |
| Ethyl enanthate..... | 0.872 ¹⁵ / ₄ | 368.8 | 187.1 | sol. | insol. |
| Other constituents: | | | | | |
| Acetal..... | 0.825 | 216 | 102.2 | ∞ | 6.25°C |
| Acetone..... | 0.792 | 133.7 | 56.5 | ∞ | ∞ |
| Pyridine..... | 0.982 | 239 | 115 | ∞ | ∞ |

* Measured at $\frac{68^{\circ}}{39.2^{\circ}}$ F, or $\frac{20^{\circ}}{4^{\circ}}$ C, unless otherwise stated.

† In 100 parts of solvent.

‡ ∞ Miscible in all proportions.

§ Approximately.

¶ Varies from 159 to 179.

Sources of data:

Lange, N. A. Handbook of chemistry. 1265 p. (See especially p. 228-461.) Handbook Publisher Inc., New York, N. Y. 1934.

Hodgman, C. D. Handbook of chemistry and physics. 23d ed. 2221 p. Chemical Rubber Publishing Co., Cleveland, Ohio. 1939.

Note: No attempt has been made to search the recent literature for later data on the physical constants nor have we tried to reconcile conflicting data. The results in parentheses are the more important divergent results reported.

present, as well as products derived from the grapes, formed in wine during aging, or liberated from the yeast cells and grape residues. (See p. 16 to 19.) From these the alcohol can only be separated by a process of fractional distillation or rectification. The volatile by-products of fermentation vary greatly in boiling point (table 12) from acetalde-

TABLE 13
SOREL'S VALUES FOR THE RATIO k FOR DIFFERENT SUBSTANCES

| Alcohol in boiling liquid | For amyl alcohol* (B. pt. 132°) | For ethyl formate (B. pt. 54.3°) | For methyl acetate (B. pt. 56°) | For ethyl acetate (B. pt. 77.1°) | For ethyl isobu- tyrate (B. pt. 110°) | For ethyl isoval- erate (B. pt. 134.3°) | For isoamyl acetate (B. pt. 137°) | For isoamyl isovaler- ate (B. pt. 196°) | For ethyl alcohol (B. pt. 78°) |
|------------------------------------|---|--|---|--|--|--|---|--|--|
| <i>Volume per cent</i> | <i>ratio</i> | <i>ratio</i> | <i>ratio</i> | <i>ratio</i> | <i>ratio</i> | <i>ratio</i> | <i>ratio</i> | <i>ratio</i> | <i>ratio</i> |
| 95 | 0.23 | 5.1 | 3.8 | 2.1 | 0.95 | 0.8 | 0.55 | 0.30 | 1.0037 |
| 90 | 0.30 | 5.8 | 4.1 | 2.4 | 1.1 | 0.9 | 0.6 | 0.35 | 1.02 |
| 85 | 0.32 | 6.5 | 4.3 | 2.7 | 1.2 | 1.1 | 0.7 | 0.40 | 1.05 |
| 80 | 0.34 | 7.2 | 4.6 | 2.9 | 1.4 | 1.3 | 0.8 | 0.50 | 1.08 |
| 75 | 0.44 | 7.8 | 5.0 | 3.2 | 1.8 | 1.5 | 0.9 | 0.65 | 1.12 |
| 70 | 0.54 | 8.5 | 5.4 | 3.6 | 2.3 | 1.7 | 1.1 | 0.82 | 1.17 |
| 65 | 0.65 | 9.4 | 5.9 | 3.9 | 2.9 | 1.9 | 1.4 | 1.05 | 1.23 |
| 60 | 0.80 | 10.4 | 6.4 | 4.3 | 4.2 | 2.3 | 1.7 | 1.30 | 1.30 |
| 55 | 0.98 | 12.0 | 7.0 | 4.9 | — | — | 2.2 | — | 1.39 |
| 50 | 1.20 | — | 7.9 | 5.8 | — | — | 2.8 | — | 1.50 |
| 45 | 1.50 | — | 9.0 | 7.1 | — | — | 3.5 | — | 1.63 |
| 40 | 1.92 | — | 10.5 | 8.6 | — | — | — | — | 1.80 |
| 35 | 2.45 | — | 12.5 | 10.5 | — | — | — | — | 2.02 |
| 30 | 3.00 | — | — | 12.6 | — | — | — | — | 2.40 |
| 25 | 5.55 | — | — | 15.2 | — | — | — | — | 2.70 |
| 20 | — | — | — | 18.0 | — | — | — | — | 3.30 |
| 15 | — | — | — | 21.5 | — | — | — | — | 4.10 |
| 10 | — | — | — | 29.0 | — | — | — | — | 5.10 |

*From fermentation.

Source of data:

Sorel, E. *Le distillation*. Paris, France. 1895.

Cited by: Monier-Williams, G. W. *Power alcohol*. 323 p. Henry Frowde and Hodder and Stoughton, London, England. 1922.

hyde, 69.8° F (21° C), to *n*-heptyl alcohol, 349.3° F (176.3° C), and the esters of acids such as caproic and enanthic boil at still higher temperatures. When a weak alcoholic wash is distilled, the volatile by-products are carried over with steam and appear with the alcohol in the distillate. When a crude spirit of high alcoholic strength is distilled, only constituents of relatively low boiling point pass over with the alcohol, while the higher-boiling constituents, the fusel oils, remain, for the most part, in the liquid.

The relative volatility of different substances from solutions of varying alcoholic strength was investigated by Sorel, and his data form the basis of modern alcohol distillery practice, particularly as interpreted

by Barbet.⁵⁹ Reilly and Hickenbottom⁶⁰ studied the distillation rate of alcohols from solutions of varying strengths and confirmed the work of Sorel. Randall and Longtin and Carey and Lewis⁶¹ have made important contributions to the theory and practice of separation processes.

Sorel's Ratio.—The data obtained by Sorel for the ratio (k) between the concentration of a substance in the vapor and in the liquid for several substances are given in table 13.

Barbet has introduced a "coefficient of purification" calculated from the k values of Sorel. This is the ratio $\frac{k_1}{k}$ where k_1 is the concentration ratio for a secondary product under consideration and k that for ethyl alcohol. The secondary constituents accumulated in the liquid at the point where $\frac{k_1}{k} = 1$. Thus, according to Monier-Williams:

If a crude spirit containing 30 per cent by volume of ethyl alcohol be boiled, the vapor will contain three times as much amyl alcohol and 2.4 times as much ethyl alcohol as the liquid. The ratio

$$\frac{k_1}{k} = \frac{3.0}{2.4} = 1.25,$$

i.e., greater than unity, and the amyl alcohol tends to accumulate in the more volatile fractions, corresponding to the foreshots of the pot still. In a liquid containing 43 per cent of alcohol by volume,

$$\frac{k_1}{k} = \frac{1.7}{1.7} = 1.0$$

and no separation of amyl alcohol from ethyl alcohol takes place. At an alcoholic strength of 70 per cent by volume,

$$\frac{k_1}{k} = \frac{0.54}{1.17} = 0.46$$

i.e., less than unity, and amyl alcohol will accumulate in the less volatile fractions, or "feints." Similarly in the case of ethyl acetate the vapor at all alcoholic concentrations will contain more ethyl acetate than the liquid, and this substance will therefore always appear in the first runnings.⁶²

As shown above, the relative volatility of the secondary constituent with respect to alcohol determines the concentration of alcohol at which

⁵⁹ Barbet, É. *A La rectification et les colonnes rectificatrices en distillerie*. 206 p. E. Bernard & Cie., Paris, France. 1895.

⁶⁰ Reilly, J., and W. J. Hickenbottom. The concentration and purification of alcoholic fermentation liquors. I. The distillation in steam of certain alcohols. Royal Dublin Society Scientific Proceedings 16:233-47. 1921.

⁶¹ Randall, Merle, and Bruce Longtin. Separation processes. *Industrial and Engineering Chemistry*, industrial edition 31:1181-86, 1295, 1299. 1939.

Carey, J. S., and W. K. Lewis. Studies in distillation. Liquid-vapor equilibria of ethyl alcohol-water mixtures. *Industrial and Engineering Chemistry*, industrial edition 24:882-83. (See especially tables 1 and 2.) 1932.

⁶² Monier-Williams, G. W. Power alcohol. 322 p. (See especially p. 75.) Henry Frowde and Hodder and Stoughton, London, England. 1922.

it will accumulate. Thus amyl alcohol is less volatile than ethyl alcohol in a strongly alcoholic solution, while in a more aqueous solution the reverse is true, and ethyl alcohol is less volatile than amyl. At an alcoholic strength of 40 to 45 per cent by volume, amyl alcohol (one of the chief constituents of fusel oil) and ethyl alcohol volatilize in the same proportions as those in which they exist in the liquid.

Separation in the Still.—In a continuous column still, the amyl alcohol tends to concentrate at the plates on which there is a solution of from 40

TABLE 14
ANALYSES OF CUTS FROM INDIVIDUAL PLATES OF A COLUMN STILL

| Plate | Initial boiling temp. | Proof | Acidity | Extract | Ash | Esters | Aldehydes* | Furfural | Higher alcohols |
|---------|-----------------------|---------|----------|---------|--------|----------|------------|----------|-----------------|
| | °F | degrees | per cent | p.p.m. | p.p.m. | p.p.m. | p.p.m. | p.p.m. | p.p.m. |
| 4..... | 174.6 | 187.1 | 0.0024 | 26 | 14 | 422.4 | 17.6 | Nil | 440.0 |
| 7..... | 175.4 | 183.5 | .0036 | 14 | 2 | 264.0 | 22.0 | Trace | 2,288.0 |
| 10..... | 176.8 | 171.3 | .014 | 24 | 2 | 1,108.8 | 30.8 | 12.5 | 15,100.8 |
| 12..... | 178.9 | 163.3 | .029 | 26 | 0 | 2,164.8 | 44.0 | 29.0 | 27,209.6 |
| 14..... | 183.0 | 134.6 | .139 | 78 | 20 | 8,430.4 | 79.2 | 54.0 | 65,560.0 |
| 16..... | 198.9 | 19.3 | .420 | 342 | 134 | 11,897.6 | 44.0 | 15.0 | 8,304.0 |
| 18..... | 206.0 | 7.9 | .216 | 796 | 170 | 3,555.2 | 30.8 | Trace | 1,056.0 |
| 20..... | 206.3 | 7.6 | .102 | 274 | 116 | 844.8 | 17.6 | Trace | 704.0 |
| 22..... | 206.4 | 7.4 | .047 | 202 | 44 | 352.0 | 13.2 | Trace | 528.0 |
| 24..... | 206.4 | 7.4 | .035 | 150 | 6 | 281.6 | 13.2 | Trace | 352.0 |
| 26..... | 206.6 | 7.3 | .030 | 140 | 2 | 211.2 | 13.2 | Nil | 528.0 |
| 28..... | 206.7 | 7.2 | .034 | 152 | 38 | 211.2 | 13.2 | Nil | 274.0 |
| 30..... | 206.6 | 7.4 | .041 | 154 | 58 | 299.2 | 8.8 | Nil | 440.0 |
| 32..... | 206.3 | 9.6 | 0.264 | 13,928 | 1,944 | 211.2 | 8.8 | Trace | 352.0 |

* Includes furfural.

Source of data:

Wilkins, Edgar A., and Margaret E. Walling. Quality brandy through controlled distillation. *The Wine Review* 7(9):7-9, 27. 1939.

to 50 per cent alcohol, and ethyl isobutyrate at 55 to 70 per cent alcohol, according to the data presented by Barbet.⁶³ In practice under California conditions, it is observed that one group of higher alcohols collects on the plate having a concentration of about 37 per cent alcohol and another at 68-75 per cent alcohol. Differences in construction and operation of stills influence these values. Data on the distribution of alcohol and congeners along a column still during operation are given in table 14.

METHOD OF DISTILLATION

The method of distillation largely determines the composition of brandy prepared from a given wine. Ordineau⁶⁴ found that the chief

⁶³ Graphically presented in Robinson, C. S., and E. R. Gilliland. *The elements of fractional distillation*. 3d ed. 267 p. (See especially p. 71-80.) McGraw-Hill Book Company, Inc., New York, N. Y. 1939.

⁶⁴ Ordineau, G. The nature of the esters of brandy and the factors which influence their proportion. *Seventh International Congress of Applied Chemistry* 8C:224-28. 1909.

factors which influence the quantities of esters in brandy are: (1) the quantity of volatile acids existing in the wine, (2) the duration of distillation, and (3) the total acidity of the wine. In his experiments, brandies distilled in a pot still with a slow fire were richer in esters than those distilled at a more rapid rate. The longer time of heating favored the esterification of the volatile acids, as was illustrated by the distillation of a mixture of neutral spirit and the residue from a previous distillation, which yielded 40 grams of esters per hectoliter of alcohol. The esters are much more readily volatile than the acids.

In a Pot Still.—Graham,⁶⁵ in studying the pot-still distillation process, found that esters and aldehydes concentrated in the early fractions, the proportions falling off as distillation proceeded, more markedly for the esters than for the aldehydes. Volatile acids were not markedly separated during the distillation. The rate of distillation influenced the relative compositions of the distillate fractions. When distillation was rapid, the alcoholic strength of the early fractions decreased very slowly, but the early fractions were comparatively rich in volatile acids. With slow distillation, concentration of the esters in the early fractions was less marked, and volatile acids tended to accumulate in the "tails." The volatile acidity of the original wine appears to have little effect on the ester content.

Proof of Distillation.—The extent of concentration of alcohol by distillation and the degree to which the nonalcohol constituents must be removed depends on the purpose for which the brandy is to be used and the nature of the congeners. In the preparation of beverage brandy, there is ordinarily no need to obtain an alcoholic strength beyond 80 to 85 per cent by volume—160° to 170° proof—for it has in any case to be diluted before consumption. At alcoholic concentrations below this range, however, the proportion of congeners increases and the brandy is heavier-bodied and requires a longer period of aging before reaching maturity.

When the distilling material is not clean, or with certain stills, distillation to over 85 per cent of alcohol by volume for beverage brandy is not uncommon. Although the resulting product is too neutral, it will be less contaminated with the undesirable aroma.

Fortifying brandy is usually distilled to 90 to 94.5 per cent by volume—180° to 189° proof. Excessive amounts of aldehydes and higher alcohols are particularly objectionable in brandies to be used for fortifying and should be as completely removed as is possible. For certain types of

⁶⁵ Graham, W. G. Influence of distillation methods on brandy composition. *Australian Brewing and Wine Journal* 58(6):40-42; (7):31-33; (8):26-31. 1940. (See review in *Institute of Brewing Journal* 46:326. 1940.)

dessert wines—muscatel, for example—however, removal of all of the flavoring constituents from the fortifying brandy is, of course, objectionable. (See also Bulletin 651, p. 87.)

TYPES OF STILLS

Pot Stills.—The simplest alcohol still used in the production of beverage brandy in California consists of a closed copper vessel, bricked in so that a fire can be built under it and known as the still (*chaudiere*); a cap (*chapiteau*); pipe (*col de cygne*); and a worm condenser (*serpentin*)

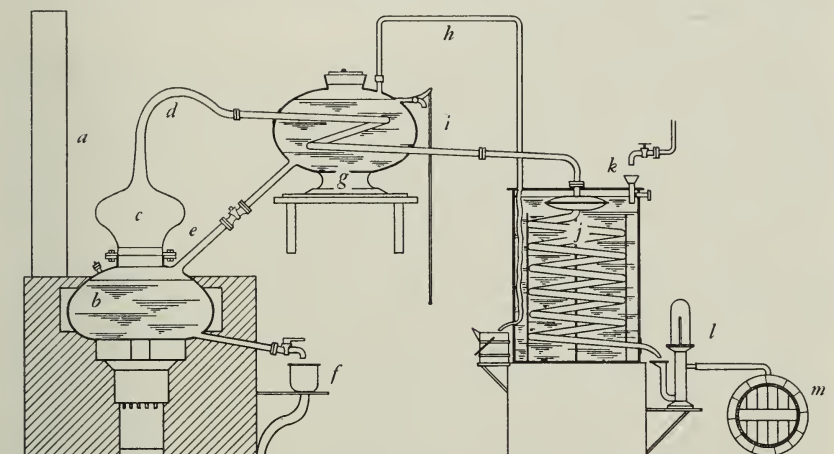


Fig. 1.—Diagram of a pot still (b) with a preheater (g) and worm condenser (j). Other parts shown are the chimney (a), cap (c), pipe (d), pipe for delivering preheated wine to the still (e), drain for removing dealcoholized wine (f), vent pipe for removing alcohol vapors from the preheater should it get too hot (h), overflow pipe for filling the preheater (i), water faucet for the condenser (k), simple try box (l) and barrel for receiving the brandy (m).

(fig. 1).⁶⁶ When the wine in the still boils, the vapor passes through the cap and pipe into the worm, a coil of pipe set in a tank filled with water in which the vapor condenses. At first the distillate contains a large percentage of alcohol; the percentage gradually diminishes; and when the distillate contains no alcohol, the distillation is stopped. The product (*brouillis*) of the first distillation usually has only 30 to 40 per cent alcohol and contains a considerable amount of aldehyde and higher alcohols. This brandy, generally termed "singlings," is purified by re-

⁶⁶ Anonymous. Brandy distillation. Appendix A to the Biennial Report of the Board of State Viticultural Commissioners for 1891-92. 125 p. California State Printing Office, Sacramento, Calif. 1892.

For a historical account of the origin of the still see:

Fairley, T. Notes on the history of distilled spirits, especially whisky and brandy. *The Analyst* 30:293-306. 1905.

Egloff, G., and C. D. Lowry, Jr. Distillation methods, ancient and modern. *Industrial and Engineering Chemistry* 21(10):920-23. 1929.

distillation. The original product may sometimes be redistilled two or three times to obtain a marketable brandy. In the second distillation the lower-boiling constituents, chiefly aldehydes, distill over with the first runnings ("heads," or foreshots), the next fraction is the clean spirit; and the last runnings ("tails," or feints) contain most of the amyl alcohol and other substances of high boiling point.

Even in the 1890's in California, these primitive stills were being supplanted by modified stills equipped with doublers and pans for the continuous redistillation of the first distillate in order to obtain a marketable brandy in one operation.

The modern pot still consists of a steam-heated kettle or boiler in which the wine to be distilled is placed. The vapors are passed through a small rectifier or fractionating column and dephlegmator into the condenser where they are condensed. The singling, if sufficiently clean, should not be redistilled because redistilling reduces the ester content of the brandy and also reduces the influence of the destructive distillation of lees, from which the process obtains its advantage. This decomposition of the lees, especially in direct-fired stills, produces important flavoring constituents, according to Valaer (cited in footnote 20, p. 10).

In brandy making by the pot still, the character of the brandy can be varied by changing the rate of distillation, by varying the proportion of "heads" and "tails," and by redistillation in the presence of an increasing proportion of "heads" from a previous run. The addition of both the "head" and "tail" fractions to a succeeding charge of wine or distilling material is not so desirable as the addition of heads alone.

Although steam is used for heating in most of the modern pot stills, the direct-fired stills are considered to give the best product.

Continuous Stills.—The separation of alcohol from water and the removal of other volatile constituents is generally accomplished by vaporizing the mixture by the suitable application of heat, condensing the vapors in such a way that fractions of varying boiling points are obtained, revaporizing these fractions and separating their vapors into similar fractions, combining fractions of similar boiling points, and repeating until the desired degree of separation is finally obtained. Although this could be done by repeated condensation and redistillation in a simple pot still, the process would be costly of fuel, time, and labor.

In the earliest type of still for the production of alcohol of high strength in one operation, the vapor from the still was bubbled through one or more smaller vessels containing alcoholic wash. The liquor in the first of these vessels became enriched with alcohol and furnished a more concentrated alcoholic vapor to the second vessel. This in turn became

still more strongly charged with alcohol and the vapor issuing from it was then condensed. The liquor in the two vessels was allowed to flow back continuously into the main still. In the modern continuous column still, the principle is the same.

In the continuous still, the distilling material is introduced continuously at one point of the column and the desired brandy flows off at another. The still essentially consists of a distilling column, a concentrating or rectifying column, a preheater, and a condenser. In the single-column still, the Böhm still, the rectifying column is located directly above the distilling column. In the Coffey still, the rectifying column is separated from the distilling column to make it possible to recover both the fusel oil and the lower-boiling constituents. This also greatly reduces the height of the apparatus. The Barbet still consists of several columns for the purpose of segregating the aldehyde, ester, and higher-alcohol fractions, removing the aldehydes and fusel oil, and returning the esters to the alcohol in a continuous manner.

The most widely used still in California is a single-column Böhm-type still. The distilling material generally enters at from 6 to 10 per cent alcohol by volume, the brandy being taken off at such a percentage as the distiller may desire, but usually at from 90 to 95 per cent by volume when it is to be used for fortifying brandy. In addition to the continuous output of brandy, there is a constant discharge of the dealcoholized distilling material, generally called "slops." Heat is usually supplied by the admission of free steam into the bottom chamber of the still. Stills of this type are usually from 36 to 60 inches in diameter and 30 to 40 feet high. (See fig. 2.) Supplementary to the distilling column, there is a dephlegmator and a condenser. The dephlegmator is placed higher than the top of the main column so that the total height of a modern continuous brandy still is often above 50 feet.

The column contains a number of perforated plates upon which the vapor is partially condensed. The perforations allow the passage of the vapor upwards through the column but they are so constructed or operated that the plates contain a certain amount of condensed liquid through which the alcoholic vapor must pass on its way to the top of the column. Bubble-cap plates (fig. 3) may be used instead of the perforated plates and are preferred by some operators. The height of the liquid on the perforated plate is regulated by the pressure of upward-flowing steam and vapors, and on the bubble-cap plate by the height of the overflow pipe. The depth of liquid on the plate is usually $1\frac{3}{4}$ inches. As more liquid is formed by condensation, it flows down to the next lower plate and eventually finds its way back to the bottom of the still. Each

plate in the column can be looked upon as a small still, and the greater the number of horizontal plates the greater the number of redistillations occurring within the still column. The vapor, as it ascends, becomes continually richer in alcohol, and the liquid in the course of its descent to

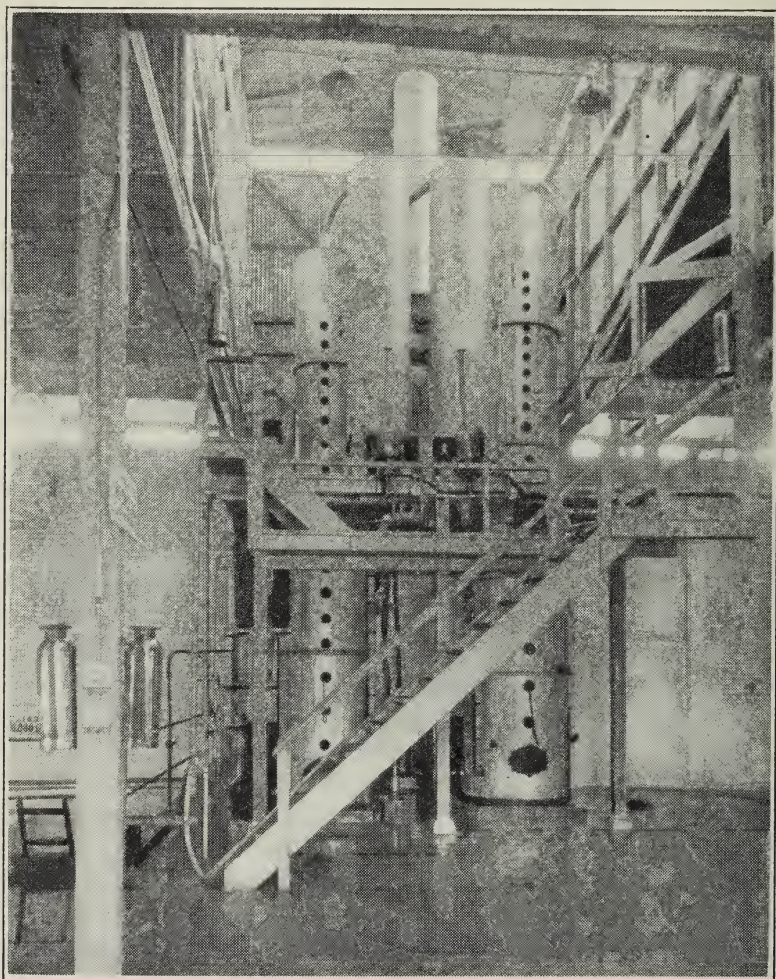


Fig. 2.—Column stills in a California winery. Note the try boxes and four fire extinguishers. (Photograph by courtesy of the Wine Institute.)

the bottom of the still gradually becomes weaker. The total number of horizontal plates varies with the design of the still, but good engineering practice for stills capable of making brandy up to 190° or 192° proof (95 to 96 per cent by volume) requires not less than forty to forty-four plates.

A transfer of material and an interchange of heat results from the contact of the vapor with a previously condensed portion; a greater enrichment of the vapor in the more volatile components is thus secured than would be possible with a single distillation using the same amount of heat. The column must be so constructed that the liquid and the vapor are in intimate contact and in equilibrium with each other at each plate. The condensed vapors returned to the column are known as the "reflux," and that portion of the column in which vapors from a still on their way to a condenser can flow counter to the portion of the condensate returned as a reflux is known as a "rectifying column." The greater the amount of reflux, other things being equal, the more nearly will the composition of the reflux approach that of the vapor entering the bottom of the column and the greater the fractionation in the column.

The height of a fractionating column or the number of plates required decreases as the ratio of reflux to distillate increases. Conversely, the reflux ratio necessary decreases as the number of plates increases.

The number of plates required also varies with the strength and purity of product desired. Comparatively few plates are required to produce a brandy of moderate strength. To produce an alcohol of high strength, not only must the number of plates be greatly increased, but also the column must be altered in design and operation.

The fractionating column should be completely insulated against heat losses to the outside to avoid inefficient utilization of the heat contained in the vapors. For efficient fractionation the reflux should be formed entirely in the condenser and returned through the full length of the

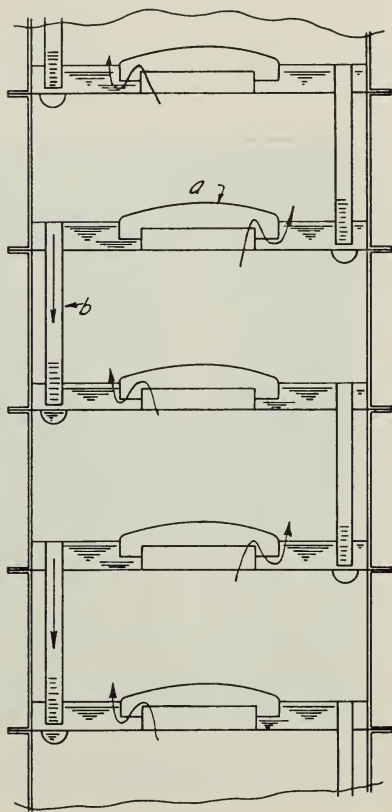


Fig. 3.—Cross section of a column still showing the bubble cap (*a*), and the overflow pipe for brandy condensing on the plate (*b*). The arrows indicate the path of vapor up the column and the course of liquid down the column.

column; this is difficult to achieve without insulation. In uninsulated columns not only is there inefficient fractionation, but also uncontrolled variations in operation occur as a result of variable losses to the surroundings. These effects are more pronounced in columns of small diameter than in those of large diameter and during intermittent than during continuous operation. Insulation of columns is not practiced widely because of the relatively high cost of insulating material. Location of the still in or near the boiler room reduces heat losses.

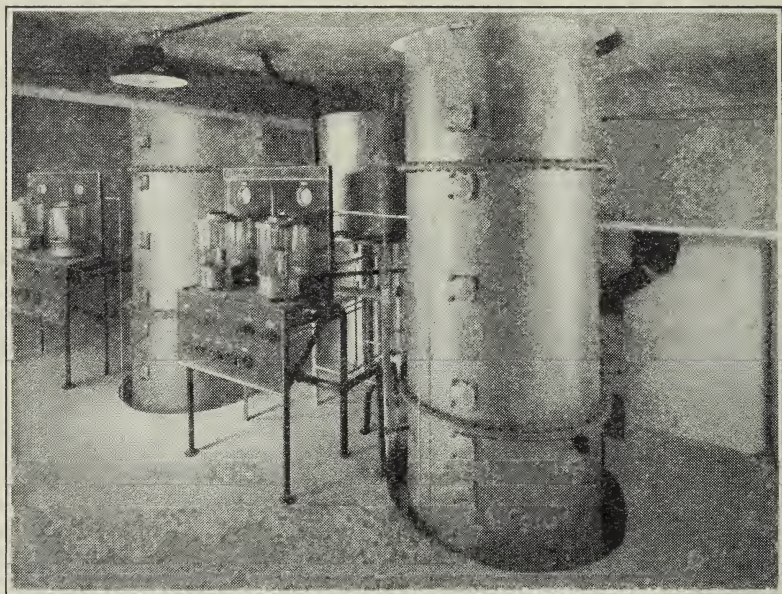


Fig. 4.—Try boxes and operating panel boards for two large continuous stills. Note the “unlagged” (noninsulated) condition of the stills. (Photograph by courtesy of the Wine Institute.)

In modern forms of rectifying still, the rectifying column is generally surmounted by a “dephlegmator,” a device in which the mixed vapor is cooled to a definite temperature, which condenses a portion of the vapor richer in higher-boiling constituents than the original vapor. The dephlegmator is essentially a partial condenser whose function is to condense a portion of the rising vapors and return the condensate to the column as reflux. It serves to economize space and makes it possible to reduce greatly the height of the rectifying column.

The rate of flow, temperature, and composition of distilling material; the rate of flow and temperature of condensation water; and the rate of flow and pressure of the steam must be controlled for optimum per-

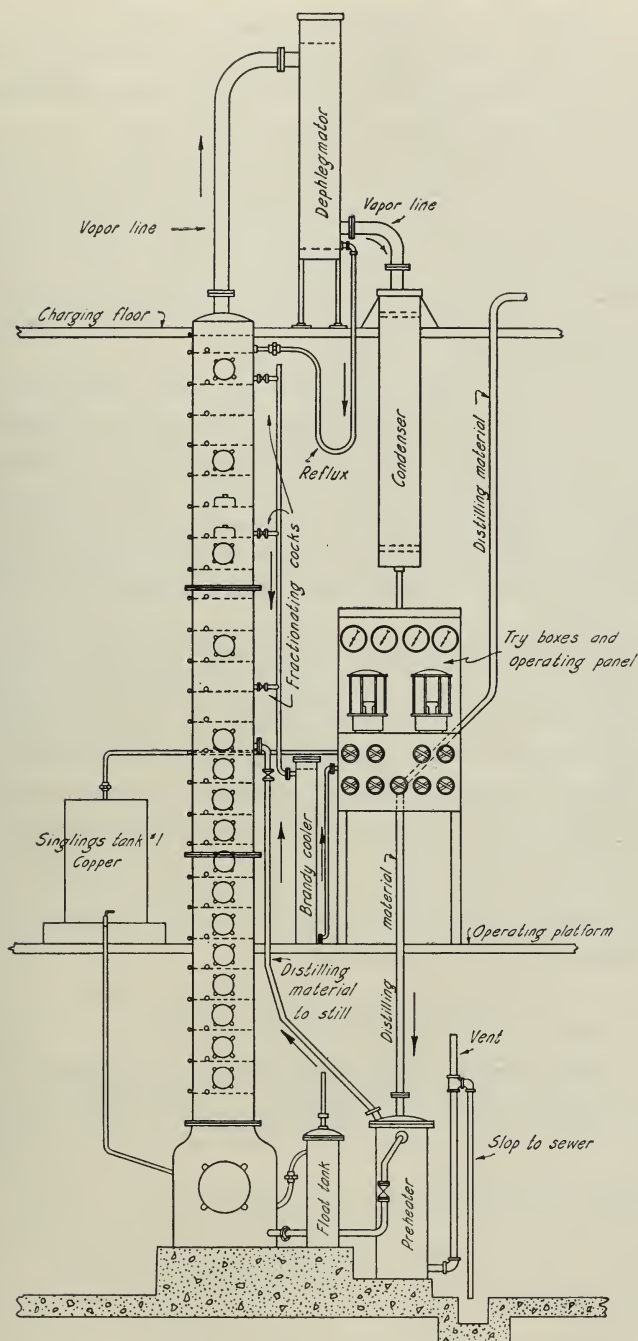


Fig. 5.—Modern column still.

formance. Automatic controlling and recording instruments are available for the larger stills.⁶⁷ Try boxes, and indicating and controlling instruments and valves should be located together on a readily accessible instrument board to facilitate operations. (See fig. 4.)

In the operation of stills, a small change in either the rate of flow or composition of the feed may not be noted in the try box (fig. 4) for from 15 minutes to several hours afterwards, the time depending upon the size of the still. By using a dephlegmator of sufficient size, it is possible to control the conditions in the column by changing the volume of reflux liquid returned to the still. This can be used to compensate for slight fluctuations in feed, water, or steam.

The size of still, the number of plates, and other items must be calculated for each definite set of conditions. There are no simple general formulas for still design. Even the calculation of the number of plates is tedious, complicated, and largely empirical. The more complicated the solution to be distilled, the more empirical are the calculations. Even the best-designed still must be tested for its performance under practical operating conditions. Typical California still assemblies are shown in figures 2 and 5.

Distilling-material preheaters are not widely enough used in California, although their use would result in definite saving in steam. The columns are rarely if ever insulated, and too many of the older stills are inadequately equipped with temperature controllers and flow meters.

The distillery premises should be constructed of fireproof materials. Smoking or open fires in the vicinity of the still should be prohibited in order to reduce the fire hazard.

Recently emptied brandy barrels and storage containers are filled with highly inflammable and asphyxiating alcohol fumes. Particular care should be exercised in handling them.

DISTILLERY OPERATION⁶⁸

DIRECTIONS FOR MAKING BEVERAGE BRANDY

For beverage purposes the wine should not be distilled at over 170° proof and preferably in a pot still, so operated that the "head" and "tail" fractions are removed.

In a Column Still.—It is difficult, with the present federal restric-

⁶⁷ Boruff, C. S., and L. P. Weiner. Advances in modern distillery operations. American Institute of Chemical Engineers Transactions 33(1):1-10. 1936.

Cummings, Ben H., and Walter S. Richert. Still building and operation. The Wine Review 5(7):6-8. 1937.

⁶⁸ General references on this subject in addition to those given in specific footnotes are listed on p. 76.

tions, to remove aldehydes and higher alcohols from brandy distilled in a continuous still, particularly of the single-column type, unless the proof of distillation is very high. If a continuous still is used for making beverage brandy, the beverage brandy should be distilled before fortifying brandy is made and in a freshly cleaned still from which all the higher alcohols and debris have been removed.

The wine is diluted to an alcoholic strength for which the column still is adjusted, usually 6 or 7 per cent, and the quantity of distilling material gauged under the supervision of the gauger. The distilling material is pumped into the charge tank located at the top of the distillery and from this it is run, usually by gravity through a float-valve controller, into the still. The plate level at which the charge enters varies but is usually between the sixth and the twelfth plate. Live steam is introduced at the bottom of the column, the condenser water turned on, and distillation begun. The first distillate is returned to the still until the proof, as indicated in the try box, is constant at the desired degree.

The condensate is then run into the brandy-receiving tanks and diluted to about 102° proof under the supervision of the gauger. The water used for reducing the proof must be pure and free from off-odors, petroleum oils, and particularly from iron. Distilled water should be used if satisfactory well water is unavailable. If compressed air is used to stir the brandy and water, it should be free from oil and rust particles.

The distillation must be conducted so as to obtain the maximum bouquet, grape flavor, and other desired characteristics with the minimum of off-flavors and -tastes due to undesired constituents. The proof of the distillate varies from 155° to 175°. Brandy distilled at higher proof is increasingly neutral, low in desired congenics, and approaches ordinary alcohol (neutral spirits) in its general qualities. Such a brandy rarely improves much on aging although it is more palatable initially. Brandies with high congeneric content require a longer aging period.

Sulfur dioxide may be removed from the brandy during distillation by inserting a small vent at the top of the column and either discarding the sulfur dioxide and other volatile sulfur compounds or percolating the vapors through a column packed with a strong oxidizing agent like permanganate.⁶⁹

It is claimed that aldehydes may be continuously removed from brandy by passing the distillate through a percolator filled with carbon and other chemical oxidation agents.⁷⁰

⁶⁹ Permission from the United States Bureau of Internal Revenue must be obtained for using these or other such processes.

⁷⁰ Fiach, Stuart M. Percolation aids brandy quality. *The Wine Review* 5(12): 14-15. 1937.

In some installations the brandy from the condenser is allowed to run through a column of oak shavings, and in others the new brandy is treated with activated carbon in a closed system, filtered, and passed through a column of oak shavings. Continuous hydrogenation of freshly distilled spirits during distillation has been advocated as a means of improving flavor.⁷¹ (See p. 60, however, on desirability of quick aging.)

After Distillation.—The receiving tanks for newly distilled beverage brandy should be glass-, copper-, or tin-lined in order to avoid discoloration of the brandy when its proof is reduced to about 102°. The corrosiveness of brandy is greater the higher its aldehyde content, and, according to Frisselle,⁷² aldehyde is more corrosive to iron than fusel oil, acetic acid, or sulfur dioxide. The beverage brandy, after reduction in proof, is usually colored with caramel sirup. The amount of caramel sirup required is predetermined by the distiller; usually 4 fluid ounces are added to a 50-gallon barrel. This produces a depth of color of 6 to 12 in the 1/2-inch cell of the Lovibond tintometer (brown series 52); and this amount of caramel coloring furnishes at the same time a total solids content of 56 to 78 grams per 100 liters of brandy. Only good-quality caramel sirup free from iron impurities and which does not impart an objectionable green color to the brandy should be used. It must be completely soluble in the brandy at 110° proof.

The brandy is barreled in weight-gauged, tared, white-oak barrels. New plain barrels of well-seasoned, no. 1 grade white oak are preferred by most California producers. Barrels made of green wood should not be used because green wood imparts off-flavors. The wood must also be dry because staves of high moisture content retard aging.⁷³ Charred barrels should not be used because they impart to brandy the oily characteristic taste of whiskey. Barrels may be re-used, but the new cooperage is preferable when the brandy is to be sold after a short storage period. Whether or not the increased extraction of woody flavors and tannin from such cooperage for quality brandies is desirable is a debatable point, but for short aging of ordinary brandies they are satisfactory. Used oak containers or soaking and steaming the barrels, would, of course, reduce this extraction of color, tannin, and flavors by the brandy but would increase the period required for aging.

Barrels for brandy storage are not usually conditioned because of the soakage allowance permitted when dry, new cooperage is used.

⁷¹ Hochwalt, Carroll A., Charles Allen Thomas, and Ernest C. Dybdal. Hydrogenation of freshly distilled spirits. *Industrial and Engineering Chemistry*, industrial edition 27:1404-7. 1935.

⁷² Unpublished data obtained by Parker Frisselle, Jr., of California Products Co., Fresno.

⁷³ Brown, E. M. The production of brandy. *The Wine Review* 6:8-10, 34. 1938.

Storage.—The brandy after filling into barrels is weighed and stored in a dry brandy warehouse under the supervision of a gauger. Withdrawals of tax-free brandy for analytical or other purposes are carefully regulated (see p. 54).

PRODUCTION OF FORTIFYING BRANDY

In the production of fortifying brandy in a continuous column still, the still is regulated to yield a distillate of about 185°–189° proof. It is usually received in galvanized-iron brandy-receiving tanks. From these it is removed to the fortification room.

Fusel oil cannot be discarded without tax payment unless permission is obtained (see p. 54), and then it may be discarded into the sewer only under the supervision of the gauger. In the operation of the still, the aldehyde and fusel-oil fractions are commonly returned to the column and accumulate there. Periodically these fractions are withdrawn into locked storage tanks. In some stills, however, provision is made for the continuous removal of the fusel oils into such tanks during operation. To permit of efficient operation this necessitates the use of two singlings tanks.

The higher alcohols can be removed from the column only when the still is washed out. In cleaning the still the alcohol present in the column is removed by distilling until the liquid in the try box just turns milky. To get rid of the singlings and fusel oil, they may be run into the column just before washing and washed down into the sewer. The still should be thoroughly washed with water and all plates and connections well cleaned. Treatment with a hot caustic solution is desirable. All reflux lines must be freed from obstruction and all vapor openings cleaned. Frequent washing and cleaning is particularly necessary with single-column stills having no provisions for continuous higher-alcohol removal.

To obtain good yields of alcohol from the distilling material, care should be taken to conduct the distillation so that all residual alcohol is removed from the slops. Only alcohol-free slops should be discharged. Their alcohol content should be determined periodically by distilling and determination of the alcohol in the distillate. An experienced still operator can detect alcohol by smell. The presence of appreciable alcohol can be detected by its effect on the hydrometer reading. The slops should not have a Balling reading of 0 or below (after correction for temperature).

PRODUCTION OF POMACE BRANDY

In making grappa, or pomace, brandy, a pot still is preferable although the new type of column pomace still has been used for this pur-

pose. Grappa brandy is sometimes stored in paraffined containers but it may acquire a paraffin taste from some containers of this type. It is never colored with caramel.

FEDERAL LAWS AND REGULATIONS

Tax.—At the present time (June, 1941), all distilled spirits except brandy are taxed at the rate of \$3.00 on each proof gallon (or wine gallon when below proof). Brandy is subject to a tax of \$2.75 a gallon.⁷⁴ This tax is a first lien on the spirits, and severe penalties are prescribed for failure to pay it. Fortifying brandy used for fortifying wines is exempt from the tax; the wines, however, are subject to certain taxes. Before any operation of a distillery may begin, the prospective distiller must register his still with the United States Bureau of Internal Revenue, give the Bureau notice of intention to engage in the business of a distiller, and finally furnish a bond, the amount of which will, in general, depend on the size of the distillery.⁷⁵ The regulations concerning distillery construction and operation are so complex that only the bare essentials can be mentioned here. The distiller should therefore operate in close conjunction with the agents of the Bureau of Internal Revenue, who are familiar not only with the multitudinous regulations but also with the interpretations of them which have been made from time to time. Much time and legal difficulty will be saved by setting up the distillery and operating it strictly according to regulations. The Wine Institute is also able to give advice about the necessary steps.

The Storekeeper-Gauger.—The operation of stills, and the measurement of the quantity of spirits produced and of the spirits needed for fortification are some of the processes in the winery handled by the storekeeper-gauger. He is an agent of the Bureau of Internal Revenue, and amicable relations between this person and the winery can do much to lighten the burden of satisfying the numerous government regulations. Particularly important is his promptness in coöperating with the winery in making the necessary tests and weighings rapidly and efficiently. A delay of 2 or 3 hours in pumping fortifying brandy into the fortification tanks may make the difference between a good and a poor wine.

Construction.—The primary intent of the regulations concerning the distillery premises are that they must be separated from other buildings

⁷⁴ United States Bureau of Internal Revenue. Production of brandy, Regulations 5. 194 p. United States Government Printing Office, Washington, D. C. 1940.

United States Bureau of Internal Revenue. Production of distilled spirits, Regulations 4. 187 p. United States Government Printing Office, Washington, D. C. 1940.

Effective October 1, 1941, the rate will be \$4.00 per proof gallon, or per wine gallon when below proof.

⁷⁵ As a rough measure of the capacity of the still in gallons per 24 hours, the government multiplies the internal cross-sectional area of the still in square feet by 960.

by unbroken partitions of substantial construction: for example, the distillery premises must open only onto an open space and not into the winery. To comply with insurance regulations, a number of California wineries have placed their still in a building which is entirely separate from the rest of the winery and is usually some distance away from it. (See fig. 3 in Bulletin 651 and compare fig. 6.) The general intent of the specific regulations concerning foundations, floors, walls, roofs, windows, and doors is that they should be constructed so as to prevent tax

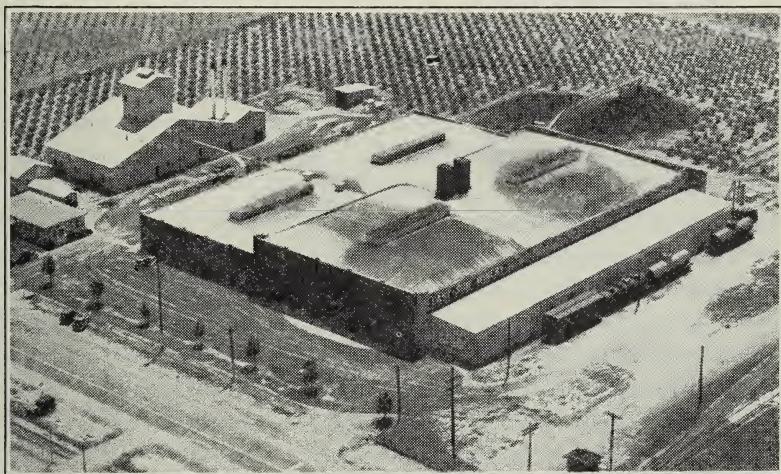


Fig. 6.—Aerial view of a large California winery. Note the distillery at the left, the tank cars at the right, and the pomace pile and vineyards in the background. (Photograph by courtesy of the Wine Institute.)

evasion either by the distiller or by some other party. The distilling-material measuring tank may be located in the distillery or winery premises.

A suitable government office is required, and in California this is usually located in the fortification room.

Equipment.—The distiller must provide a sign, giving the name of the distiller and certain other information, suitable scales, test weights, weighing tanks, and the regular equipment of a standard still. Drawings of the premises and details of the still construction are also required by law. Metal receiving tanks are generally most desirable as they can be locked directly. Measuring devices which operate by a float are permitted. Provision is made in the law for “heads” and “tails” receiving tanks in the still room. The regulations concerning removal and destruction of these substances have, in general, been considered so impractical by the industry that the tanks have not in general been used sufficiently.

Considerable improvement in the quality of the fortifying brandies particularly would result if the "heads" and "tails" were more carefully removed. The distillate should contain half of 1 per cent or more of aldehydes in the "heads" and 1 per cent or more of fusel oils in the "tails" tank. No tax is paid on spirits so removed when they are destroyed or denatured under government supervision.

Try boxes are a necessary adjunct of the still. They must be constructed so as to permit reading the proof and temperature without unlocking. (See fig. 4, p. 46.) Unfortunately, however, it is not only the proof at the try box which is important, but also the quality of the spirits, as well as the proof and quality of the spirits on each of the upper plates of the stills. The strict government regulations regarding the operation of the still and the taking of the samples no doubt handicap the distiller in operating the still to produce the best brandy or to secure the highest yield. Although provisions are made for the removal of one pint or smaller samples from the still for laboratory examination, the difficulty of securing the samples is sufficiently onerous to prevent most operators from using the privilege.

Labeling.—The fermenters and other tanks are required to be properly labeled with their purpose and size. All pipes in the still room must also be painted: the prescribed colors for pipes are black for brandy or finished spirits; blue for vapor, singlings, high or low wines; red for distilling material; brown for slop; gray for fermenting material; yellow for fusel oil; white for water; aluminum for steam; and orange for air.

Rectification.—If spirits are purified, refined, or rectified after the distillation, the process is known as "rectification," and the spirits so treated are subject to a rectifying tax. The person purifying, refining, or rectifying the spirits must pay the special federal occupational tax of a rectifier and perform these operations on premises completely separated from the distillery.

But the spirits may be variously treated during an original and continuous distillation. So long as the process is not discontinuous, the distiller may run the spirits through as many processes of distillation as he wishes. In certain industrial-alcohol plants two or three stills in series are used to produce the types and qualities of spirits required.

The law also permits distillers to purify or refine distilled spirits in the course of the original and continuous distillation. Processes so approved are percolation through oak chips (the oak chips must not have been treated with any chemical and must be approved by the United States Bureau of Internal Revenue), introduction of substances (such as potassium permanganate or hydrogen peroxide) into the brandy dur-

ing the course of the distillation for purifying the brandy, and others. Approval for the use of such processes should, of course, be obtained before use.

After Distillation.—Burnt sugar or caramel sirup should be added to the brandy prior to the time it is gauged and removed from the distillery to the warehouse. Not more than 6 fluid ounces per 50 gallons of brandy may be added. The regulations also cover the methods of gauging, and a special gauging manual (see footnote 5, p. 4) is provided to convert pounds to gallons, and so on. The packages into which the brandy is removed must be serially numbered and otherwise branded with required information—gross weight, tare weight, net weight, wine gallons, proof, and proof gallons, and other marks and brands. The brandy is usually reproofed after storage and before bottling. There is no provision for losses by leakage or evaporation on distillery premises.

Reports and Records.—Accurate and regular keeping of the necessary numerous forms is very essential. Form 1520 concerns such items as brandy produced and gauged for the month, and is one of the more important forms. When not in use, the distillery must be so registered.

Form 15 has to do with a record of distillery operations, and a duplicate is kept at the winery. The forms are usually self-explanatory, although a new distillery should obtain a full explanation from the Bureau of Internal Revenue or from some experienced storekeeper-gauger of what is required in each form before using it.

Numerous locks are required on the still proper. But furnace doors may be locked as well as manheads, inlets, outlets, and so on. The government also requires keys to the distillery premises.

DISTILLERY-SLOP DISPOSAL

The dealcoholized and diluted solution from the still contains a considerable amount of material in solution or suspension. This material offers a difficult problem of disposal because of the large volume obtained from the still and the difficulty in decomposition of the material itself. Although distillery-slop disposal is not a health problem, the oxygen requirements for its decomposition are so great that it constitutes a hazard to fish life in stream disposal, and its decomposition by-products are volatile and highly obnoxious. It is the chief problem in the disposal of winery wastes.⁷⁰

⁷⁰ De Martini, F. E. Disposal of wastes from wineries. California Sewage Works Journal 7:47-51. 1934.

Marquis, H. H. The problem of waste disposal. The Wine Review 5(11):13-14, 23-24. 1937.

Shaw, P. A. Pollution of the Mokelumne River by winery wastes. Sewage Works Journal 9:599-606. 1937.

Shaw, P. A. Pollution control work of the California State Division of Fish and Game. Sewage Works Journal 12(5):947-53. 1940.

No winery should create an unpleasant odor near its premises. Obnoxious odors not only constitute a basis of just complaint by neighbors, but they are also a poor policy from the standpoint of the impression which the winery creates for prospective wine buyers. The odors of some distillery-slop settling basins are little short of nauseating, and for this and the reasons given above should be removed without delay.

The material from the still is composed of suspended lees material, chiefly yeasts, of the salts, acids, glycerin, and residual sugar of the wine, and—over 95 per cent—of water.

Industrial Disposal.—The most convenient method of disposal is through an industrial sewage plant either operated directly by the winery or by some municipal agency. In an industrial sewage-disposal plant, the slop is treated as sewage and decomposed and disposed of in the most expeditious manner. Although the municipality may make an extra charge for use of its plant by the winery, in general this is the best and cheapest method for disposal of distillery slop and winery wastes. In the beer industry the basis of the charge by the municipality is generally the oxygen requirement for decomposition of a representative sample of brewery waste or the amount of suspended solids.

If there is no municipal sewage-disposal plant available, the winery may have to build a plant. This, however, is expensive and should be installed only after a study of all alternatives has been made.⁷⁷

The basic problem is to separate the large volume of water from the suspended material. After that, the effluent water no longer contains objectionable organic matter and can be disposed of in streams or may simply be run into basins. The lees and other residues are then handled by themselves.

Several methods of separating the suspended material and the water are available, such as chemical treatment, sedimentation, and trickle filters.

In chemical treatment some flocc-forming chemical is added to the slop, usually an aluminum or ferric salt and occasionally lime, and a precipitate is formed. The clear supernatant liquid is then removed and the sludge handled as described on page 57.

In sedimentation the liquid and suspended solids are allowed to flow through settling tanks. These tanks may be equipped with mechanical devices for removing the sludge and skimming the surface. The sludge and effluent liquid are handled as indicated on page 57.

In a trickle filter the slop is sprayed into a bed of rock and gravel.

⁷⁷ Busbee, R. J. Disposal of brewery wastes. Wallerstein Laboratories Communications 3(8):18-29. 1940.

Buswell, A. M., and N. Le Bosquet. Complete treatment of distillery wastes. Industrial and Engineering Chemistry, industrial edition 28:795-97. 1936.

A bacterial flora develops on the rock and removes some of the organic matter and makes the rest more stable and easier to settle. The material from the bottom of the bed is removed and the water separated from the sludge in a settling tank.

The sludge from any of the above procedures must still be decomposed, but it is separated from the volume of liquid and can be handled much more expediently, since its volume is only about one-twentieth that of the original material. In California this sludge can be dried in open basins, and if placed on the beds to a depth of only a foot or less, will dry quickly. The surface is then loosely cultivated and a new sludge run in. A better practice is to build a digestion tank. The sludge is placed in digestion tanks where anaerobic bacteria grow, and after a period of a month the sludge is so decomposed that it may be dried in open beds without the development of obnoxious odors.

Australian Process.—In Australia, Hodgson and Johnston⁷⁸ have worked out a complete industrial disposal system for a winery. Their regular municipal sewage plant was found to fail to function properly when distillery slop was run through in large amounts. A pretreatment plant was therefore constructed for the winery alone. The steps in their process included precipitation with 1,200 parts per million of lime, flocculation and sedimentation, dilution of the settled waste with plant effluent, adjustment of the pH to 7.0–8.0 with acid, treatment of the liquid in trickle filters in two stages, with a humus tank after the first stage. The residual material was then sent to the regular municipal sewage-disposal plant (an activated sludge type). Because of the possibility of odor nuisances, the flocculation tanks and trickle filters were enclosed. The trickle filters also operated under a forced draft.

In the design and construction of such plants, the services of a sanitation expert should be engaged. Pilot plant experiments are very useful.

Basin Treatment.—The usual method of distillery-slop disposal used in California, and the most objectionable and undesirable, is to run the material into settling basins. Usually too small an area is provided and the slop soon accumulates. Decomposition sets in and foul and nauseating odors are produced.

The settling-basin method can be made to operate without undue accumulation of odors if a very large area is provided so that the slop does not accumulate, or if the area is subsoiled and is sufficiently porous to remove large volumes of liquid. In a few cases the winery is fortunate enough to have a large area for basins in a district where winter floods remove the deposited slop.

⁷⁸ Hodgson, H. J. N., and J. Johnston. The treatment of winery wastes at Glenelg, South Australia. *Sewage Works Journal* 12:321–40. 1940.

The possibility of contaminating the underground water supply with distillery slop must also be borne in mind. Proebsting and Jacob⁷⁹ have shown that toxic principles are either present in the slop, or more probably are produced or brought into solution from the soil during the decomposition of the slop. The toxicity was sufficient to kill plants when distillery slop was used as irrigation water and also the soil fertility was injured. For these reasons, distillery slop should not be used for irrigation nor allowed to settle into an underground water supply which may be used as a source of water for drinking, bathing, or irrigation purposes.

By-product Utilization.—Because of the large amount of yeast cells found in the slop, it has sometimes been flocculated, filtered, and dried. Busbee reports such wastes from breweries are about as high in nitrogen content as ordinary barnyard manure. Schulz,⁸⁰ on the contrary, has shown various distillery wastes to be of restricted value for feed or fertilizer purposes.

Cruess and Weast⁸¹ have suggested the possible utilization of the winery distillery slop as a source of vitamins. The study has not been extended sufficiently, but their results showed little B present. In whiskey distillery slop, Boruff, Langlykke, and Black⁸² found a high content of riboflavin.

Distillery slop from pot stills which is not diluted with steam condensate is a convenient source of cream of tartar, if the original wine contained much cream of tartar. This recovery is less feasible from the continuous stills used in California, and particularly from slops derived from the distillation of the low-acid wines which are produced by washing the pomace.

PREPARATION FOR MARKET⁸³

Fortifying brandies are not usually aged, and only beverage brandies are regularly stored. The purposes of aging are to allow the spirits to extract tannin, color, flavoring materials, and other substances from the wood, and to permit the brandy to undergo oxidation, esterification, and other chemical changes which will make it more drinkable. (See p. 20 to 23.)

⁷⁹ Proebsting, E. L., and H. E. Jacob. Some effects of winery distillery waste on soil and plants. *American Society for Horticultural Science Proceedings* 36:69-73. 1938.

⁸⁰ Schulz, A. G. Ueber den Geldwert der Schlempe. *Brennerei Zeitung* 49:201. 1932.

⁸¹ Cruess, W. V., and C. Weast. The utilization of pomace and still slops. *The Wine Review* 8(2):10-13, 27-28. 1939.

⁸² Boruff, C. S., A. F. Langlykke, and S. Black. Vitamin content of distillers' by-products. *Industrial and Engineering Chemistry, industrial edition* 32:1237-39. 1940.

⁸³ General references on this subject in addition to those given in specific footnotes in the section are listed on p. 76.

BRANDY STORAGE AND AGING

The conditions of storage, as well as the treatment of brandy after distillation, are closely regulated by the United States Bureau of Internal Revenue. After barreling and weighing, the brandy is stored in a locked warehouse which may be entered only in presence of a gauger. (See fig. 7.)



Fig. 7.—Brandy aging in oak barrels in a California winery.
(Photograph by courtesy of the Wine Institute.)

At the time of removal from the Bureau of Internal Revenue bonded warehouse, the brandy is regauged and the tax is paid on the spirits remaining, provided (*a*) that the brandy must be regauged before it is eight years old, and (*b*) that the losses do not exceed those listed in table 15.

The barrels in storage should be examined periodically and leaky staves replaced.

Rapid Aging.—The quick aging of brandies is discouraged by the federal agencies and by the usually undesirable results obtained with the permissible treatments: filtration through activated carbons, oxidation with hydrogen peroxide or other oxidizing agents, addition of oak chips, and heating. The addition of plain untoasted white-oak chips of high quality, carefully controlled as to amount, is usually beneficial when the brandy is to be stored *less than four years*. About 1 gram of oak

TABLE 15
MAXIMUM ALLOWABLE LOSSES FOR LEAKAGE AND EVAPORATION OF
BRANDY STORED IN AN INTERNAL REVENUE BONDED WAREHOUSE

| Time in storage | | Maximum allowance* | Time in storage | | Maximum allowance* |
|-----------------|---------------|----------------------|-----------------|---------------|----------------------|
| More than | Not more than | | More than | Not more than | |
| <i>months</i> | <i>months</i> | <i>proof gallons</i> | <i>months</i> | <i>months</i> | <i>proof gallons</i> |
| .. | 2 | 1.0 | 33 | 36 | 7.5 |
| 2 | 4 | 1.5 | 36 | 40 | 8.0 |
| 4 | 6 | 2.0 | 40 | 44 | 8.5 |
| 6 | 8 | 2.5 | 44 | 48 | 9.0 |
| 8 | 10 | 3.0 | 48 | 52 | 9.5 |
| 10 | 12 | 3.5 | 52 | 56 | 10.0 |
| 12 | 15 | 4.0 | 56 | 60 | 10.5 |
| 15 | 18 | 4.5 | 60 | 64 | 11.0 |
| 18 | 21 | 5.0 | 64 | 69 | 11.5 |
| 21 | 24 | 5.5 | 68 | 72 | 12.0 |
| 24 | 27 | 6.0 | 72 | 76 | 12.5 |
| 27 | 30 | 6.5 | 76 | 80 | 13.0 |
| 30 | 33 | 7.0 | 80 | .. | 13.5 |

* For containers of 40 or more gallons. Containers of from 20 to 40 gallons' capacity are permitted losses up to 50 per cent of these. The maximum allowable losses are measured from the date of the original gauge in the case of brandy but in other spirits it is measured from the date of original entry into the warehouse.

Source of data:

United States Bureau of Internal Revenue, Regulations 10, Warehousing of distilled spirits, 189 p. (See especially p. 78.) U. S. Government Printing Office, Washington, D. C. 1940.

chips per liter of brandy is sufficient. Heating hastens the formation of volatile esters but is usually unnecessary in California brandy warehouses where high summer temperatures are recorded. Artificial heating of warehouses, the preheating of brandy in the barrel by use of immersion heaters (steam coils or electric heaters), and even storage in hot rooms for a few days has been practiced. Brown⁸⁴ finds that a uniform storage temperature of 70° to 80° F is most favorable to aging.

Type of Container.—The commercial brandy prepared in California is usually stored under bond in white-oak barrels for aging (fig. 8). There is, however, some difference of opinion as to the pretreatment of

⁸⁴ Brown, E. M. Brandy improvement after distillation. The Wine Review 5(6): 15-16. 1937.

the barrels. The French authorities recommend a particular type of oak barrel (Limosin oak) and state that storage in these containers is particularly responsible for the quality of cognac brandies. French white-oak barrels are also sometimes used by other European countries for the storage of brandy. In order to determine the effect of kind of treatment of barrel and to test various agents, a number of 50-gallon lots of brandy were placed under observation in 1934.⁸⁵ Samples of the brandy



Fig. 8.—Brandy barrels in storage racks. Note the elevator for raising the barrels and the gauger testing the proof. (Photograph by courtesy of the Wine Institute.)

were withdrawn periodically and brought to the laboratory for analyses. After withdrawal they were stored at 32° F in glass bottles until analyzed.

The analyses after storage for one year are shown in table 16. During this period of aging there was a somewhat erratic variation in tannin content, a definite increase in iodine value, extract, and volatile acidity, and an erratic increase in acetaldehyde content with the length of storage in cask. The original brandy was very raw and harsh in flavor. Storage for two weeks had very little effect on improvement of flavor. Three months later there was an appreciable smoothing and mellowing;

⁸⁵ The California Barrel Company and the Western Cooperage Company furnished the barrels and the Roma Wine Company furnished the brandy used.

and after seven months and two weeks, the improvement was marked, some of the brandies being palatable.

In the tests on effect of treatment of barrels, the untreated barrel proved best. Steaming or washing with soda ash removed too much of the extractives so that the brandy still remained raw after one year of aging. Hypochlorite treatment gave a marked off-flavor. Charring gave a brandy of whisky flavor although fairly smooth. The brandy in the paraffined barrel was not only raw but had a taste of paraffin.

TABLE 16
ANALYSES OF BRANDY STORED IN BARRELS FOR ONE YEAR

| Type and treatment of barrel and substance added | Extract | Tannin | Iodine (0.01 N) | Total acid as acetic | Volatile acid as acetic | Aldehyde |
|--|------------------|------------------|-----------------|----------------------|-------------------------|----------|
| | grams per 100 cc | grams per 100 cc | cc per 100 cc | grams per 100 cc | grams per 100 cc | p. p. m. |
| Paraffined second-grade oak..... | 0.062 | 0.0024 | 5.0 | 0.0246 | 0.022 | 88.0 |
| Charred new white oak..... | .112 | .0031 | 9.2 | .0432 | .033 | 93.0 |
| Steamed new white oak..... | .106 | .0031 | 7.4 | .0422 | .033 | 88.5 |
| Sodium-hypochlorite-treated new white oak..... | .089 | .0003 | 4.6 | .0420 | .033 | 92.2 |
| Soda-ash- and steam-treated new white oak..... | .089 | .0020 | 6.0 | .0425 | .033 | 94.0 |
| Untreated new white oak: | | | | | | |
| No substances added..... | .219 | .0080 | 4.8 | .0444 | .033 | 87.0 |
| 2 oz. quercyl added..... | .119 | .0143 | 8.2 | .0456 | .032 | 70.8 |
| 8 oz. Shaco BB oak chips added..... | .093 | .0035 | 6.0 | .0432 | .035 | 68.0 |
| 8 oz. white oak chips added..... | .097 | .0052 | 9.6 | .0468 | .029 | 66.5 |
| Cognac oak extract added..... | .094 | .0038 | 8.6 | .0480 | .037 | 74.5 |
| Tannol added..... | 0.144 | 0.0035 | 8.4 | 0.0432 | 0.030 | 65.6 |

Of the treatments used, the addition of white-oak chips was most beneficial and quercyl was next best. The others were of little benefit, charred (Shaco BB) oak chips giving an unpleasant flavor. After one year of aging, the best brandies were those stored in untreated white-oak barrels with added white-oak chips, with added quercyl, and in plain barrels, in about that order.

Valaer (cited in footnote 20, p. 10) has made many analyses of brandy and of the changes occurring in brandy during aging. In his experiments on the effect of the kind of container, he obtained results similar to those above.

Excessive extraction of oak constituents may occur when the brandy is stored for a long time in new oak barrels, particularly in presence of added oak chips. Steamed or used barrels are, therefore, probably preferable for brandies to be aged for over five years.

Fortifying Brandy.—In most cases the fortifying brandies produced

in California wineries are used entirely by the winery itself. Occasionally they must be moved from one winery to another or stored for use the next season. Iron drums, galvanized-iron drums, and drums coated with some protective substance, such as "Amercoat," are used for shipping or holding fortifying brandy. Concrete tanks have also been successfully used for storage, but they must be carefully constructed if they are to be used for this purpose. If metal containers are used for storage, the spirits must be free of sulfur dioxide. Storage of spirits high in this substance in iron cooperage not only leads to contamination of the spirits, but is also very injurious to the container itself. In certain cases such spirits have had to be redistilled before use.

TASTING

The tasting of brandies is more difficult than that of wines. Unless only one or two samples are to be tasted, the brandy should always be diluted before tasting, usually one to one with distilled water. Because of the very dehydrating effect of brandies on the palate, especial importance should be given to the odorous constituents so as to reduce the oral examination as much as possible. Glasses which have a large bowl and a small opening are useful for smelling brandies—these are sometimes called "brandy sniffers." The glass should not be too thick so that the heat from the hand may quickly warm the liquid and help to volatilize the more aromatic principles. Many tasters, however, prefer a somewhat smaller tasting glass which does not accentuate the aroma so much. It is even advantageous sometimes to briskly rub a little of the undiluted brandy between the palms of the hands and quickly smell in order to detect slight off-aromas.

Fortifying brandies are tested entirely by smell. They should be free of excess fusel oil. If the brandy clouds on dilution with distilled water, it is usually considered that the concentration of higher alcohols is too great. The fortifying brandy should be clean-smelling and free of all pomace odor.

The organoleptic examination of distilled spirits is sometimes made on samples obtained by fractional distillation of the spirit. It is said that the character of certain fractions is a useful method of determining sophistication, or adulteration. It is true that it is difficult to determine sophistication by analysis, but organoleptic detection of it may also be difficult.⁸⁶

⁸⁶ For a long argument of the relative merits of analysis and organoleptic examination in the detection of adulteration in brandies see: Buttner, C., and A. Miermeister. *Beiträge zur Untersuchung und Beurteilung von Weindestillaten und Weinbränden. Zeitschrift für Untersuchung der Lebensmittel* 56:492-98; 1928. 58:628-35; 1929.

See also: Zellner, H. *Beiträge zur Untersuchung und Beurteilung von Weindestillaten und Weinbränden. Zeitschrift für Untersuchung der Lebensmittel* 59:431-33. 1930.

BLENDING

Government regulations do not permit blending of brandies except on rectifying premises but do exempt the blended brandy from the rectifying tax under certain conditions. There is thus unfortunately very little opportunity for the distiller to improve his product by rational blending of brandies of different ages and types. About the only purpose of brandy tasting then is to segregate the brandies produced into various grades for storing different periods of time and bottling under different labels. Modification of the brandy blending regulations would help distillers to improve the quality of their product.

BOTTLING

When brandy is diluted with water to lower alcohol proof before bottling, part of the insoluble caramel constituents and oak extractives



Fig. 9.—Glass containers used for bottling brandy: *a*, screw-cap bottle used for California beverage brandy; *b*, *c*, *d*, cork-closure bottles used by cognac shippers; *e* and *f*, front and side views of flask used for armagnac brandy; *g*, side view of half-pint, and *h*, front view of pint flask used for distilled spirits.

precipitate immediately. Another part of the constituents rendered insoluble by the decrease in alcoholic content will precipitate more slowly. This is due largely to the slowness with which the colloidal particles which form at first increase in size. The initial alcoholic content, storage temperature, the amount of caramel, the nature and concentration of oak extractives, and the extent of dilution, as well as other factors, influence the rate at which equilibrium is reestablished. The acidity of the brandy markedly influences this rate: the higher the acidity and the lower the pH value, the faster this deposition. To avoid the formation of reddish-brown precipitates in the bottled brandy, the aged brandy after cutting with water should be stored for several days before final filtration. The federal authorities now allow the storage of diluted brandy in a closed container for two or three days. Although this period is sufficient for most brandies, it may not be long enough for some very

old brandies which contain higher concentrations of oak extractives. It has been suggested that the rate of addition of distilled water may affect the cloudiness and flavor of brandy. Slow addition is the best procedure. Glass-lined or stainless-steel containers are preferable for storage of brandy prior to bottling.

Brandies bottled after they reach four years in the wood may be bottled in bond, but the government requires the distiller to use a special stamp giving the age and stating that it has been bottled in bond.

The brandy is usually filtered before bottling. Every effort should be made to eliminate metallic contamination in the pipe lines, filters, and bottling equipment even if it is necessary to install glass tubes or other corrosion-resistant conduits.

The bottles used must be free of aluminum or other harmful substances which the alcohol might dissolve and which would cause undesirable precipitates to form. The usual closure is a screw cap. (See fig. 9.)

ANALYSIS OF BEVERAGE AND FORTIFYING BRANDIES⁸⁷

Both research and routine control procedures are included in this section, with particular stress on the latter. Kelly⁸⁸ has studied the methods of examination for testing commercial fortifying brandy.

ALCOHOL DETERMINATION

Proof.—The proof of spirits may be determined approximately by proof hydrometers directly, or, more accurately, by proof hydrometers or other means, on the distillate after distillation.

Pipette 25 cc of spirits into a Kjeldahl flask and add 100 cc of water. Distill nearly 100 cc into a volumetric flask, make to volume with water at the standardized temperature. Determine the specific gravity by weighing in a pycnometer. The specific gravity is corrected for weight *in vacuo* as follows:

$$G = \frac{S + 0.00105 W}{W + 0.00105 W},$$

where G is the corrected specific gravity, S is the weight of sample, and W is the weight of water. The percentage by volume may be obtained from table 17 if the pycnometer weighing has been made at 60° F, or 15.56° C.

The percentage alcohol is also conveniently obtained in the distillate by the use of an immersion refractometer. The scale readings of the instrument are converted to volume percentage of alcohol by reference

⁸⁷ General references of this subject in addition to those given in specific footnotes in the section are listed on p. 76 to 77.

⁸⁸ Kelly, C. W. Methods of examination suitable for testing commercial fortifying spirits. Australian Brewing and Wine Journal 58(3):46-47. 1939.

TABLE 17

PROOF, ALCOHOL PERCENTAGES OF VOLUME AND WEIGHT, SPECIFIC GRAVITY, SCALE READINGS ON THE REFRACTOMETER, AND INDEX OF REFRACTION OF ETHYL ALCOHOL AND WATER MIXTURES

| Proof | At 60° F (15.56° C) | | | | At 68° F (20° C) | |
|------------|-----------------------------|------------------------------|--------------------------|------------------|------------------|---------------------|
| | Alcohol, per cent by volume | Alcohol, per cent by weight* | Grams alcohol per 100 cc | Specific gravity | Scale reading | Index of refraction |
| 0.0..... | 0.00 | 0.00 | 0.00 | 1.00000 | 14.4 | 1.33296 |
| 5.0..... | 2.50 | 1.99 | 1.98 | 0.99629 | 17.5 | 1.33416 |
| 10.0..... | 5.00 | 4.00 | 3.97 | 0.99281 | 20.93 | 1.33548 |
| 15.0..... | 7.50 | 6.02 | 5.96 | 0.98959 | 24.45 | 1.33684 |
| 20.0..... | 10.00 | 8.04 | 7.93 | 0.98660 | 28.1 | 1.33824 |
| 25.0..... | 12.50 | 10.08 | 9.92 | 0.98381 | 31.9 | 1.33968 |
| 30.0..... | 15.00 | 12.13 | 11.90 | 0.98114 | 35.8 | 1.34116 |
| 35.0..... | 17.50 | 14.19 | 13.89 | 0.97859 | 39.8 | 1.34266 |
| 40.0..... | 20.00 | 16.26 | 15.87 | 0.97608 | 43.06 | 1.34421 |
| 45.0..... | 22.50 | 18.34 | 17.86 | 0.97355 | 48.0 | 1.34574 |
| 50.0..... | 25.00 | 20.43 | 19.84 | 0.97097 | 52.05 | 1.34727 |
| 55.0..... | 27.50 | 22.54 | 21.83 | 0.96828 | 56.05 | 1.34874 |
| 60.0..... | 30.00 | 24.66 | 23.81 | 0.96541 | 60.0 | 1.35022 |
| 65.0..... | 32.50 | 26.80 | 25.79 | 0.96235 | 63.8 | 1.35160 |
| 70.0..... | 35.00 | 28.96 | 27.78 | 0.95910 | 67.33 | 1.35291 |
| 75.0..... | 37.50 | 31.14 | 29.76 | 0.95560 | 70.6 | 1.35409 |
| 80.0..... | 40.00 | 33.35 | 31.74 | 0.95185 | 73.6 | 1.35518 |
| 85.0..... | 42.50 | 35.58 | 33.73 | 0.94786 | 76.4 | 1.35622 |
| 90.0..... | 45.00 | 37.84 | 35.71 | 0.94276 | 79.1 | 1.35719 |
| 95.0..... | 47.50 | 40.13 | 37.69 | 0.93916 | — | — |
| 100.0..... | 50.00 | — | 40.40† | 0.9344 | — | 1.35805† |
| 105.0..... | 52.50 | — | 42.65 | 0.9295 | — | — |
| 110.0..... | 55.00 | — | 45.15 | 0.9244 | — | 1.3596 |
| 115.0..... | 57.50 | — | 47.65 | 0.9190 | — | — |
| 120.0..... | 60.00 | — | 50.10 | 0.9136 | — | 1.3612 |
| 130.0..... | 65.00 | — | 55.21 | 0.9021 | — | 1.3623 |
| 140.0..... | 70.00 | — | 60.49 | 0.8900 | — | 1.36334 |
| 150.0..... | 75.00 | — | 65.93 | 0.8773 | — | 1.3641 |
| 160.0..... | 80.00 | — | 71.56 | 0.8639 | — | 1.3648 |
| 170.0..... | 85.00 | — | 77.48 | 0.8496 | — | 1.36495 |
| 180.0..... | 90.00 | — | 83.83 | 0.8339 | — | 1.3643 |
| 190.0..... | 95.00 | — | 90.70 | 0.8161 | — | 1.36345 |
| 200.0..... | 100.00 | — | 98.52 | 0.7939 | — | — |

* Grams per 100 grams.

† Data from Handbook of Chemistry to 40.0 per cent weight; thereafter from International Critical Tables and at 68° Fahrenheit instead of 60° Fahrenheit.

‡ Data from Handbook of Chemistry to 45.00 per cent volume; thereafter from International Critical Tables. Figures in italics from interpolated data.

Sources of data:

Hodgman, C. D. Handbook of chemistry and physics. 18th ed. 1,818 p. Chemical Rubber Publishing Co., Cleveland, Ohio. 1933.

Methods of Analysis of the Association of Official Agricultural Chemists. 5th ed. 757 p. Association of Official Agricultural Chemists, Washington, D. C. 1940.

Washburn, E. W. International Critical Tables of numerical data, physics, chemistry, and technology. Vol. III. p. 116-19. McGraw-Hill Book Co., New York, N. Y. 1928.

to table 17. The refractive index is, however, markedly affected by changes in temperature, and this table must not be used unless the reading is made at the temperature given. Complete tables giving the refractive index at various temperatures are given in the *Methods of Analysis* of the Association of Official Agricultural Chemists.

Occasionally an ebulliometer is used for certain liquids.⁸⁹ The chemical procedure for determination of alcohol in wines has been previously mentioned in Bulletin 651.

Methyl Alcohol.—Fruit brandies normally contain small quantities of methyl alcohol. (See p. 10.) It is determined by preferential oxidation to formaldehyde, which is then estimated colorimetrically with Schiff's reagent. Beyer⁹⁰ has developed a colorimetric procedure using a neutral wedge photometer. (A photoelectric colorimeter may be used also.)

ACID DETERMINATION

Total Acid.—Neutralize about 250 cc of boiled water in a large porcelain evaporating dish or in a 400-cc beaker. Add 25 cc of sample and titrate to a faint pink, using 0.1 *N* NaOH and 2 cc of phenolphthalein solution as the indicator. In brandies the total acid is usually expressed as acetic. The cubic centimeters of NaOH used multiplied by 0.024 gives grams of acid as acetic per 100 cc of spirit. Occasionally the substances in the brandy derived from the wood and the added caramel turn orange-brown near the end point. An outside indicator may then be useful.

Fixed Acid.—Evaporate 25 cc to dryness in a small porcelain dish (or preferably in a platinum dish) on a steam bath and dry about 1 hour in an oven at 100° C (212° F). Dissolve and transfer the residue to a large porcelain dish with 25 cc of neutral alcohol. The alcohol should be about the same proof as that of the sample. Add 250 cc of neutralized boiled water and 2 cc of phenolphthalein solution. Titrate with 0.1 *N* NaOH. In young brandies the fixed-acid content is very small. A 10-cc microburette or the use of more dilute NaOH is recommended whenever the fixed-acid content is low.

The fixed acid is calculated in the same manner as the total acid.

Volatile Acid.—The total-acid content minus the fixed-acid content gives the amount of volatile acid.

ESTER DETERMINATION

Pipette exactly 100 cc of brandy and about 12½ cc of water into a 200-cc distillation flask. Distill slowly nearly 100 cc into a 100-cc volu-

⁸⁹ Love, R. F. A table for ebulliometers for use with alcoholic liquids containing solid matters. *Industrial and Engineering Chemistry*, analytical edition 11:548-50. 1939.

⁹⁰ Beyer, G. F. Application of neutral wedge photometer to the quantitative determination of methanol in distilled spirits. *Association of Official Agricultural Chemists Journal* 22:151-56. 1939.

metric flask. The last 25 cc should be distilled at a maximum heat to assure distillation of all of the furfural. The receiving tube from the condenser should reach well into the receiving flask. Electric heat is preferable to gas, because local overheating may result when gas is used and some furfural may be produced. Bring the distillate to volume at the calibrated temperature. Take 50 cc of the distillate in a round-bottom flask and neutralize the free acid present with 0.1 *N* NaOH, using phenolphthalein as an indicator. This titration is sometimes considered to represent the volatile acids present, but not all the volatile acids distill by this procedure and this therefore represents only a minimum value for the volatile acids. Then add a measured excess amount of 0.1 *N* NaOH, usually 25 cc. Connect the flask to a reflux condenser and boil for 1 hour. Cool the flask and titrate with 0.1 *N* HCl. The cubic centimeters of excess 0.1 *N* alkali added minus the cubic centimeters of 0.1 *N* HCl used for the final titration gives the cubic centimeters of 0.1 *N* alkali required to saponify the esters present. The number of cubic centimeters required multiplied by 17.6 gives the milligrams of ester as ethyl acetate in 100 cc of spirits. An occasional blank using water in place of distillate should be run and the necessary correction applied. Tobie⁹¹ has shown that aldehydes do not materially interfere with the ester determination when this procedure is used. They do apparently interfere when the saponification is allowed to take place at room temperatures over a 24-hour period.

ALDEHYDE DETERMINATION

Acetaldehyde.—Place 100 cc brandy in a 500-cc distilling flask, acidify with 20 cc of a 5 per cent phosphoric acid solution, dilute to 150 cc (add 3–4 pieces of capillary glass tubing, sealed at one end, to prevent bumping), and distill over 100 cc. Collect the distillate in a 500-cc flask containing 50 cc of a neutral buffer solution (3.35 grams of KH_2PO_4 and 15 grams of $\text{Na}_2\text{HPO}_4 \cdot 12 \text{H}_2\text{O}$ per liter—the water used for diluting these salts should be boiled to prevent growth of molds) and 10 cc of a bisulfite solution (18.9 grams of anhydrous sodium sulfite and 150 cc of *N* sulfuric acid per liter). Before distilling, mark the receiving flask at 160 cc. Place the delivery tube below the surface of the liquid. Distill until the 160-cc mark is reached.

Stopper, shake, and let the 500-cc flask stand 20 minutes. Add 1 cc of freshly prepared 0.2 per cent starch solution, 100 cc of water, and 10 cc of acid solution (1 part of concentrated HCl to 3 parts of water). Then titrate the excess bisulfite with 0.1 *N* iodine solution.

⁹¹ Tobie, W. C. Improved methods of distilled-liquor analysis. Food Research 6(1):15–29. 1941.

Add 100 cc of alkaline solution (8.75 grams of boric acid and 400 cc of *N* sodium hydroxide per liter, approximately 16.5 grams C. P. NaOH) and titrate with 0.01 *N* iodine to determine the liberated sulfite. The sulfite liberated at this pH is considered to be that combined with aldehydes and not oxidized by the first titration with iodine in the acid solution.

The number of cubic centimeters of iodine $\times 2.2$ gives the number of milligrams of acetaldehyde in a liter.

This is essentially the Jaulmes and Espezel procedure. The recovery of acetaldehyde by this procedure is about 90 per cent.

Furfural.—The basis for this method is the color developed by furfural in the presence of aniline and hydrochloric acid.

Prepare a standard furfural solution by weighing out 1 gram of re-distilled furfural and diluting to 100 cc with 95 per cent alcohol. This solution is fairly stable. Also prepare a dilute furfural solution using 1 cc of the standard and diluting to 100 cc with 50 per cent alcohol. One cc of this latter solution contains 0.1 mg of furfural. The aniline used must be as nearly colorless as possible. It can be prepared by re-distillation.

An aliquot of the distillate prepared for the ester determination may be used for this procedure. Dilute 10 to 20 cc of this distillate to 50 cc with 50 per cent alcohol in a large test tube. Also dilute 0.5, 1, 2, 3, and 4 cc of the dilute furfural standard with 50 per cent alcohol to 50 cc in large test tubes. Add 2 cc of colorless aniline and 0.5 cc of HCl (sp. gr. 1.125) to all the tubes. Mix and leave in a water bath at about 60° F for 15 minutes. Compare the color in the unknown with that of the standards, either directly or with one of the knowns in a color comparator. A photoelectric color comparator may be used advantageously.

Acetal.—From 15 to 30 per cent of the aldehyde in brandy is combined with alcohol as the aromatic acetal. The principal source of aldehyde and of acetal is the oxidation of alcohol occurring during the aging of brandy in wood. Peynaud and Maurie⁹² report from 86 to 185 mg of aldehyde per liter of cognac and from 64 to 94 mg of acetal per liter. They suggest the following method of determining acetal based on the rapid dissociation of acetal in hot acid solutions into free aldehyde (at pH 2) in comparison with its stability at pH 9.

Distill 50 cc of brandy and 25 cc of a buffer solution at pH 9 (25 grams of sodium borate and 25 cc of *N* H₂SO₄ dissolved in 1 liter of distilled water) into a 50 cc volumetric flask containing 25 cc of distilled water. After mixing and making to volume at the temperature at which the

⁹² Peynaud, E., and A. Maurie. L'acetal dans les eaux-de-vie, son dosage volumétrique. Reactions d'acétalisation. Bulletin International du Vin 1938:33-38.

flask is calibrated, two 20-cc portions are removed and distilled separately. To one portion add 5 cc of sirupy phosphoric acid (85 per cent) and 25 cc of water; to the second 30 cc of buffer at pH 9. Distill these into 50-cc volumetric flasks containing 25 cc of a sulfite solution neutralized to pH 7. Then determine the free aldehyde in both cases by the Jaulmes and Espezel procedure (see p. 68), using 0.02 *N* iodine solution for the second titration (1 cc corresponds to 0.44 mg of acetaldehyde). The difference between the two parallel titrations corresponds to the quantity of acetal present in 20 cc of brandy. Multiply this difference in volume of 0.02 iodine solution by 2.68 to obtain the weight of acetal in milligrams.

HIGHER-ALCOHOL DETERMINATION

The determination of higher alcohols is one of the more important procedures in brandy analysis. It is also one of the least satisfactory from the standpoint of accuracy and simplicity. Two procedures are commonly used in determining the higher-alcohol fraction of brandies. In the Allen-Marquardt procedure, which has been adopted as the official method by the Association of Official Agricultural Chemists, the higher alcohols are oxidized to the corresponding acids and subsequently estimated by titration with 0.1 *N* alkali. In the Schiektanz-Etienne acetyl chloride procedure, the higher alcohols are esterified and the degree of esterification is estimated by a titration procedure in which 0.1 *N* alkali and 0.1 *N* sulfuric acid are used. The former procedure is given in detail here, although for more accurate work the latter is preferable.⁶³

Add to 50 cc of brandy, 50 cc of water and 20 cc of 0.5 *N* NaOH and boil under a reflux condenser for 1 hour. Distill 90 cc from this, add 25 cc of water and continue until a total of 115 cc are received. (If aldehydes are present in excess of 150 parts per million, 0.5 gram of metaphenylenediamine hydrochloride should be added to the distillate; the solution refluxed 1 hour; 100 cc distilled off, 25 cc of water added, and a total of 125 cc finally received.)

Add to the distillate NaCl (sodium chloride) as salt and as a saturated solution until the specific gravity is 1.10. Extract this saline solution with 40, 30, 20, and 10 cc of pure CCl₄. Wash the CCl₄ solution three times with 50-cc portions of saturated NaCl solution and twice with saturated Na₂SO₄ solution. Be sure all NaCl is removed by these two washings. Transfer the CCl₄ to a flask containing 50 cc of a K₂Cr₂O₇

⁶³ Schiektanz, S. T., and A. D. Etienne. Determining the amyl alcohol content of distilled spirits. *Industrial and Engineering Chemistry*, analytical edition 11:390-93. 1939.

Schiektanz, S. T., A. D. Etienne, and J. L. Young. Correlation of fusel oil values by the Allen-Marquardt and acetyl chloride method. *Association of Official Agricultural Chemists Journal* 23:368-73. 1940.

(potassium dichromate) solution (prepared by dissolving 100 grams of $K_2Cr_2O_7$ in 900 cc of H_2O and adding 100 cc of H_2SO_4) and boil 8 hours under a reflux condenser.

Add 100 cc of water and distill until only about 50 cc remains. Add 50 cc of water and again distill until only 35–50 cc is left. The distillate must be water-white. Local overheating and burning during the distillation should be avoided. Tobie recommends the addition of 5 to 10 drops of 10 per cent $Na_2S_2O_3$ (sodium thiosulfate) if the distillate is a yellowish color. Titrate the distillate with 0.1 *N* NaOH using phenolphthalein indicator. One cc of 0.1 *N* NaOH equals 0.0088 gram of amyl alcohol. The higher-alcohol fraction is usually reported as amyl alcohol even though alcohols other than amyl alcohol occur in fusel oil (see p. 11).

Ground-glass connections for all apparatus are preferable, and a blank determination should occasionally be conducted on 100 cc of the CCl_4 solution, beginning the blank determination at the step where the CCl_4 is washed three times with saturated NaCl.

MISCELLANEOUS TESTS

The permanganate test originally developed by Barbet is still used in detecting impurities in high-quality alcohol. Although the nature of the permanganate-reducing materials is not known and the results are not entirely specific, it is of value in determining the extent of refining and detects the presence of excessive amounts of higher alcohols and sulfur compounds. As originally developed by Barbet, the test consisted in determining the time required to decolorize at 18° C the permanganate added to a sample which had been diluted to an alcohol content of 42.5 per cent by volume. Kelly has modified this procedure as follows: Place 50 cc of spirit, the strength of which has been adjusted to 50 per cent by volume, in a thoroughly clean flask which has a ground-glass stopper. The sample is brought to 20° C, 2 cc of a solution of potassium permanganate (containing 0.2 gram per liter) is added, and the flask stoppered and shaken to mix. The color steadily fades, and the time taken in seconds or minutes to reach that of a synthetic standard solution is recorded as the permanganate test time. With normal brandies the decolorization is almost instantaneous, "hard" or less neutral brandies require less than a minute, while highly rectified samples may take 15 minutes or more to fade to the standard color. It is therefore applicable only to rectified spirits.

The standard is a salmon-pink tint obtained by mixing 10 cc of cobalt chloride solution (25 grams per liter) and 10 cc of uranium nitrate solution (28 grams per liter) and making to 50 cc. The comparison is sufficiently accurate when made in two similar flasks. The color given by high-

quality samples usually approaches that of the standard very closely, but those giving a shorter test time are frequently of a distinctly lighter shade.

The test is not very sensitive to small variations in alcoholic strength, but is appreciably affected by temperature. The results may be completely falsified if the alcohol makes contact with foreign substances—for example, dirty glass and, especially, ordinary corks.

Barbet's original recommendations were that the alcohol should be of a strength of 42.5 per cent by volume, and the temperature 18° C, but the above small modifications do not affect relative values. These, together with a compensating adjustment of the strengths and volumes of the synthetic standard, were adopted for the sake of uniformity and convenience.

Fluorescence of fractions of the brandy when illuminated by ultraviolet light has been used in detecting adulteration of brandies.⁹⁴

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⁹⁴ Rudiger, M., and E. Mayr. Über das Verhalten alkoholischen Destillate im ultravioletten Licht. *Zeitschrift für Untersuchung der Lebensmittel* 57:561–66. 1929.

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⁹⁵ See also the footnotes in each section of the text for additional references.

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